

Search Report

EIC 1700

STIC Database Tracking Number: 239129

To: EBENEZER SACKY
Location: REM-5B31 / Mailbox 5C18
Art Unit: 1624
Thursday, October 11, 2007

Case Serial Number: 10/519513

From: KATHLEEN FULLER
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REM-4B28 / REM-4B19
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Search Notes

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SCIENTIFIC REFERENCE
Sci & Tech Inf. Ctr.
OCT 0 2 1993

Scientific and Technical Information Center

SEARCH REQUEST FORM

Requester's Full Name: BEN SACKY Examiner #: 73489 Date: 10/2/07
Art Unit: 1824 Phone Number: 2-0704 Serial Number: 10/59513
Location (Bldg/Room#): 1EM533 (Mailbox #): C-18 Results Format Preferred (circle): PAPER DISK

To ensure an efficient and quality search, please attach a copy of the cover sheet, claims, and abstract or fill out the following:

Title of Invention: Process for removing H₂O from a mixture containing

Inventors (please provide full names): Jungkamp et al.

Earliest Priority Date: 07/04/03

Search Topic:

Please provide a detailed statement of the search topic, and describe as specifically as possible the subject matter to be searched. Include the elected species or structures, keywords, synonyms, acronyms, and registry numbers, and combine with the concept or utility of the invention. Define any terms that may have a special meaning. Give examples or relevant citations, authors, etc., if known.

For Sequence Searches Only Please include all pertinent information (parent, child, divisional, or issued patent numbers) along with the appropriate serial number.

A process for removing H₂O from a mixture of H₂O and zinc chloride comprising adding a polar (aprotic) diluent to the mixture @ given an anhydrous mixture of zinc chloride and diluent, wherein the diluent consists of 2-cis-pentene-1,2-dithiol, 2-trans-pentene-1,2-dithiol etc.

Reacts

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Searcher Prep & Review Time: 40

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Type of Search

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____ AA Sequence (#)

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✓ Bibliographic

____ Litigation

____ Fulltext

____ Other

Vendors and cost where applicable

✓ STN _____ Dialog

____ Questel/Orbit _____ Lexis/Nexis

____ Westlaw _____ WWW/Internet

____ In-house sequence systems

____ Commercial _____ Oligomer _____ Score/Length

____ Interference _____ SPDI _____ Encode/Transl

____ Other (specify)

=> FILE HCAPL

FILE 'HCAPLUS' ENTERED AT 14:52:34 ON 11 OCT 2007

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FILE COVERS 1907 - 11 Oct 2007 VOL 147 ISS 16

FILE LAST UPDATED: 10 Oct 2007 (20071010/ED)

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This file contains CAS Registry Numbers for easy and accurate substance identification.

=> D QUE L33

L2 11 SEA FILE=REGISTRY ABB=ON (16529-66-1/BI OR 16545-78-1/BI OR 20068-02-4/BI OR 25899-50-7/BI OR 26294-98-4/BI OR 30574-97-1/B I OR 4786-24-7/BI OR 592-51-8/BI OR 7646-85-7/BI OR 7647-01-0/B I OR 7732-18-5/BI)

L3 1 SEA FILE=REGISTRY ABB=ON 7646-85-7

L4 1 SEA FILE=REGISTRY ABB=ON 7732-18-5

L5 8 SEA FILE=REGISTRY ABB=ON L2 AND NITRIL?

L6 51847 SEA FILE=HCAPLUS ABB=ON L3 OR ZNCL2 OR ZINC CHLORIDE

L7 2720480 SEA FILE=HCAPLUS ABB=ON L4 OR WATER?

L8 8915 SEA FILE=HCAPLUS ABB=ON L6 AND L7

L9 556 SEA FILE=HCAPLUS ABB=ON L5

L10 6 SEA FILE=HCAPLUS ABB=ON L8 AND L9

L11 96403 SEA FILE=HCAPLUS ABB=ON REMOV?(4A) L7

L12 424 SEA FILE=HCAPLUS ABB=ON L8 AND (L11 OR DEWATER?)

L13 10 SEA FILE=HCAPLUS ABB=ON L12 AND AZEOTROP?

L14 24 SEA FILE=HCAPLUS ABB=ON L12 AND ?NITRIL?

L15 2 SEA FILE=HCAPLUS ABB=ON L12 AND APROTIC?

L16 0 SEA FILE=HCAPLUS ABB=ON L12 AND HETEROAZEOTROP?

L17 35 SEA FILE=HCAPLUS ABB=ON L10 OR L13 OR L14 OR L15 OR L16

L18 5 SEA FILE=HCAPLUS ABB=ON L12 AND POLAR?

L19 37 SEA FILE=HCAPLUS ABB=ON L17 OR L18

L20 4 SEA FILE=HCAPLUS ABB=ON L19 AND INORGANIC?/SC, SX

L21 24 SEA FILE=HCAPLUS ABB=ON L12 AND INORG?/SC

L22 3039 SEA FILE=HCAPLUS ABB=ON AZEOTROPES+NT, PFT/CT

L23 1 SEA FILE=HCAPLUS ABB=ON L21 AND L22

L24 1 SEA FILE=HCAPLUS ABB=ON L12 AND L22

L25 1 SEA FILE=HCAPLUS ABB=ON L21 AND ?AZEOTROP?

L26 3 SEA FILE=HCAPLUS ABB=ON L21 AND DILUT?

L27 246011 SEA FILE=HCAPLUS ABB=ON NITRILES+NT, PFT/CT

L28 235 SEA FILE=HCAPLUS ABB=ON L8 AND L27

L29 9 SEA FILE=HCAPLUS ABB=ON L12 AND L28

L30 14 SEA FILE=HCAPLUS ABB=ON L20 OR (L23 OR L24 OR L25 OR L26) OR L29

L32 0 SEA FILE=HCAPLUS ABB=ON L21 AND DILUENT?

L33 14 SEA FILE=HCAPLUS ABB=ON L30 OR L32

=> D L33 BIB ABS IND HITSTR 1-14

L33 ANSWER 1 OF 14 HCAPLUS COPYRIGHT 2007 ACS on STN
 AN 2005:144220 HCAPLUS
 DN 143:45276
 TI Envirocat EPZ10: a recyclable solid acid catalyst for the synthesis of Biginelli-type 3,4-dihydropyrimidin-2(1H)-ones
 AU Lee, Key-Young; Ko, Kwang-Youn
 CS Department of Chemistry, Ajou University, Suwon, 443-749, S. Korea
 SO Bulletin of the Korean Chemical Society (2004), 25(12), 1929-1931
 CODEN: BKCSDE; ISSN: 0253-2964
 PB Korean Chemical Society
 DT Journal
 LA English
 OS CASREACT 143:45276
 AB A one-pot method for the Biginelli reaction was developed, using Envirocat EPZ10 as a recyclable solid acid catalyst. EPZ10 is prepared by supporting ZnCl₂ on clay and has predominantly strong Lewis acid sites and weak Bronsted acid sites. A model reaction of benzaldehyde, Et acetoacetate, and urea was carried out using 3 types of Envirocats, EPZ10, EPZG (clay-supported FeCl₃), and EPIC (clay-supported polyphosphoric acid). Envirocats catalysts were activated via azeotropic drying with toluene to remove loosely bound water in clay. In refluxing toluene, EPZ10 was the most active, with 84% product yield within 6 h vs. classical Biginelli conditions (catalyst HCl in EtOH, reflux, 18 h) with 80% yield. Reactions of aromatic and aliphatic aldehydes were carried out using EPZ10 in refluxing toluene. Envirocat EPZ10 is environmentally friendly as ZnCl₂, the active component, is nontoxic and inexpensive. The catalyst can be easily recovered by simple filtration and reused after activation by drying at 110° in air.
 CC 45-4 (Industrial Organic Chemicals, Leather, Fats, and Waxes)
 Section cross-reference(s): 28
 ST zinc chloride Envirocat EPZ10 recyclable catalyst
 Biginelli synthesis; green chem solid acid catalyst recovery activation
 Biginelli synthesis
 IT Cyclocondensation reaction
 Cyclocondensation reaction catalysts
 (Biginelli reaction; environmentally benign and recyclable Envirocat EPZ10 and EPZG and EPIC solid acid catalysts for Biginelli synthesis of dihydropyrimidinones)
 IT Solvent effect
 (aprotic vs. protic; environmentally benign and recyclable Envirocat EPZ10 and EPZG and EPIC solid acid catalysts for Biginelli synthesis of dihydropyrimidinones)
 IT Green chemistry
 (environmentally benign and recyclable Envirocat EPZ10 and EPZG and EPIC solid acid catalysts for Biginelli synthesis of dihydropyrimidinones)
 IT Clays, uses
 Polyphosphoric acids
 RL: CAT (Catalyst use); USES (Uses)
 (environmentally benign and recyclable Envirocat EPZ10 and EPZG and EPIC solid acid catalysts for Biginelli synthesis of dihydropyrimidinones)
 IT Lewis acidity
 (strong; environmentally benign and recyclable Envirocat EPZ10 and EPZG and EPIC solid acid catalysts for Biginelli synthesis of

- dihydropyrimidinones)
- IT Bronsted acidity
(weak; environmentally benign and recyclable Envirocat EPZ10 and EPZG and EPIC solid acid catalysts for Biginelli synthesis of dihydropyrimidinones)
- IT 169494-75-1, Envirocat EPZG
RL: CAT (Catalyst use); USES (Uses)
(clay supported FeCl₃; environmentally benign and recyclable Envirocat EPZ10 and EPZG and EPIC solid acid catalysts for Biginelli synthesis of dihydropyrimidinones)
- IT 174631-90-4, Envirocat EPZ 10
RL: CAT (Catalyst use); USES (Uses)
(clay supported ZnCl₂; environmentally benign and recyclable Envirocat EPZ10 and EPZG and EPIC solid acid catalysts for Biginelli synthesis of dihydropyrimidinones)
- IT 332076-74-1, Envirocat EPIC
RL: CAT (Catalyst use); USES (Uses)
(clay supported polyphosphoric acid; environmentally benign and recyclable Envirocat EPZ10 and EPZG and EPIC solid acid catalysts for Biginelli synthesis of dihydropyrimidinones)
- IT 7646-85-7, Zinc chloride (ZnCl₂),
uses 7705-08-0, Iron chloride (FeCl₃), uses
RL: CAT (Catalyst use); USES (Uses)
(environmentally benign and recyclable Envirocat EPZ10 and EPZG and EPIC solid acid catalysts for Biginelli synthesis of dihydropyrimidinones)
- IT 5395-36-8P 17994-63-7P 110448-29-8P 123629-39-0P 123629-40-3P
123629-41-4P 149990-62-5P 161374-07-8P 161374-08-9P 198826-86-7P
363138-52-7P
RL: IMF (Industrial manufacture); PREP (Preparation)
(environmentally benign and recyclable Envirocat EPZ10 and EPZG and EPIC solid acid catalysts for Biginelli synthesis of dihydropyrimidinones)
- IT 57-13-6, Urea, reactions 100-52-7, Benzaldehyde, reactions 141-97-9,
Ethyl acetoacetate
RL: RCT (Reactant); RACT (Reactant or reagent)
(environmentally benign and recyclable Envirocat EPZ10 and EPZG and EPIC solid acid catalysts for Biginelli synthesis of dihydropyrimidinones)
- IT 64-17-5, Ethanol, uses 75-05-8, Acetonitrile, uses 75-09-2,
Methylene chloride, uses 108-88-3, Toluene, uses 109-99-9, THF, uses
RL: NUU (Other use, unclassified); USES (Uses)
(reaction solvent; environmentally benign and recyclable Envirocat EPZ10 and EPZG and EPIC solid acid catalysts for Biginelli synthesis of dihydropyrimidinones)
- IT 7646-85-7, Zinc chloride (ZnCl₂),
uses
RL: CAT (Catalyst use); USES (Uses)
(environmentally benign and recyclable Envirocat EPZ10 and EPZG and EPIC solid acid catalysts for Biginelli synthesis of dihydropyrimidinones)
- RN 7646-85-7 HCAPLUS
CN Zinc chloride (ZnCl₂) (CA INDEX NAME)

Cl-Zn-Cl

- IT 75-05-8, Acetonitrile, uses
RL: NUU (Other use, unclassified); USES (Uses)

(reaction solvent; environmentally benign and recyclable Envirocat
EPZ10 and EPZG and EPIC solid acid catalysts for Biginelli synthesis of
dihydropyrimidinones)

RN 75-05-8 HCAPLUS

CN Acetonitrile (CA INDEX NAME)

 $\text{H}_3\text{C}-\text{C}\equiv\text{N}$

RE.CNT 22 THERE ARE 22 CITED REFERENCES AVAILABLE FOR THIS RECORD
ALL CITATIONS AVAILABLE IN THE RE FORMAT

L33 ANSWER 2 OF 14 HCAPLUS COPYRIGHT 2007 ACS on STN

AN 2004:60454 HCAPLUS

DN 140:130117

TI Method of in-process recycling of Lewis acids in production of nitriles by hydrocyanation

IN Jungkamp, Tim; Scheidel, Jens; Luyken, Hermann; Bartsch, Michael; Baumann, Robert; Haderlein, Gerd

PA BASF Aktiengesellschaft, Germany

SO PCT Int. Appl., 27 pp.

CODEN: PIXXD2

DT Patent

LA German

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	WO 2004007431	A1	20040122	WO 2003-EP7150	20030704
	W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW				
	RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG				
	DE 10231292	A1	20040129	DE 2002-10231292	20020710
	DE 10240012	A1	20040311	DE 2002-10240012	20020827
	CA 2491240	A1	20040122	CA 2003-2491240	20030704
	AU 2003246375	A1	20040202	AU 2003-246375	20030704
	EP 1521737	A1	20050413	EP 2003-763704	20030704
	R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR, BG, CZ, EE, HU, SK				
	BR 2003011982	A	20050426	BR 2003-11982	20030704
	CN 1665776	A	20050907	CN 2003-816115	20030704
	JP 2005538075	T	20051215	JP 2004-520479	20030704
	MX 2004PA12030	A	20050307	MX 2004-PA12030	20041202
	US 2005247624	A1	20051110	US 2004-520007	20041230
	US 7084294	B2	20060801		
	IN 2004CN02996	A	20060217	IN 2004-CN2996	20041231
PRAI	DE 2002-10231292	A	20020710		
	DE 2002-10240012	A	20020827		
	WO 2003-EP7150	W	20030704		

AB A Lewis acid is recycled from a nitrile-containing reaction mixture (I) obtained

by hydrocyanation of an olefinically-unsatd. compound, the mixture having a miscibility gap with water under certain concentration, pressure and

temperature conditions, the hydrocyanation process being carried out in the presence of a catalyst system comprising a Lewis acid and a complex of a phosphorus-containing compound as a ligand and a central atom suitable for the ligand. The recycling process comprises the steps of (a) separating the complex from the mixture (I) to obtain a mixture (II), (b) treating the mixture (II) with water at such pressure and temperature conditions that a phase (III) with a higher proportion of water than of the nitrile and a phase (IV) with a higher proportion of the nitrile than of water are formed, the phase (III) having a higher content of the Lewis acid than the phase (IV), (c) mixing the phase (III) with a liquid diluent (V) which does not form an azeotrope with water and having b.p. higher than that of water under certain pressure conditions, or which forms an azeotrope with water under certain pressure conditions. The recycling process further comprises the steps of (d) distilling the mixture of the phase (III) and the liquid diluent (V) under certain pressure conditions to obtain a mixture (VI) having a higher proportion of water than of the liquid diluent (V) and a mixture (VII) having a higher proportion of the diluent (V) than of water, the mixture (VII) having a higher content of the Lewis acid than the mixture (VI), and (e) feeding the mixture (VII) to the stage of hydrocyanation of an olefinically-unsatd. compound to produce a nitrile. The process can be used for recycling of zinc chloride in production of adipodinitrile by hydrocyanation of a nitrile mixture produced by hydrocyanation of butadiene.

- IC ICM C07C253-10
- CC 45-4 (Industrial Organic Chemicals, Leather, Fats, and Waxes)
Section cross-reference(s): 35, 67
- ST Lewis acid hydrocyanation catalyst recycling nitrile prodn
- IT Hydrocyanation catalysts
(method of in-process recycling of Lewis acids in production of nitriles by hydrocyanation)
- IT Lewis acids
RL: CAT (Catalyst use); USES (Uses)
(method of in-process recycling of Lewis acids in production of nitriles by hydrocyanation)
- IT Nitriles, preparation
RL: IMF (Industrial manufacture); NUU (Other use, unclassified); RCT (Reactant); PREP (Preparation); RACT (Reactant or reagent); USES (Uses)
(method of in-process recycling of Lewis acids in production of nitriles by hydrocyanation)
- IT Alkenes, reactions
RL: RCT (Reactant); RACT (Reactant or reagent)
(method of in-process recycling of Lewis acids in production of nitriles by hydrocyanation)
- IT Solvents
(organic; method of in-process recycling of Lewis acids in production of nitriles by hydrocyanation)
- IT 7646-85-7, Zinc chloride, uses
RL: CAT (Catalyst use); USES (Uses)
(method of in-process recycling of Lewis acids in production of nitriles by hydrocyanation)
- IT 111-69-3P, Adipodinitrile
RL: IMF (Industrial manufacture); NUU (Other use, unclassified); PREP (Preparation); USES (Uses)
(method of in-process recycling of Lewis acids in production of nitriles by hydrocyanation)
- IT 592-51-8, 4-Pentenitrile 16529-56-9, 2-Methyl-3-butenitrile
16545-78-1, cis-3-Pentenitrile 20068-02-4,
cis-2-Methyl-2-butenitrile 25899-50-7, cis-2-Pentenitrile
26294-98-4, trans-2-Pentenitrile 30574-97-1,

trans-2-Methyl-2-butenenitrile
 RL: NUU (Other use, unclassified); USES (Uses)
 (method of in-process recycling of Lewis acids in production of nitriles by hydrocyanation)
 IT 16529-66-1, trans-3-Pentenitrile
 RL: NUU (Other use, unclassified); RCT (Reactant); RACT (Reactant or reagent); USES (Uses)
 (method of in-process recycling of Lewis acids in production of nitriles by hydrocyanation)
 IT 7646-85-7, Zinc chloride, uses
 RL: CAT (Catalyst use); USES (Uses)
 (method of in-process recycling of Lewis acids in production of nitriles by hydrocyanation)
 RN 7646-85-7 HCAPLUS
 CN Zinc chloride (ZnCl₂) (CA INDEX NAME)

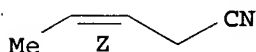
Cl-Zn-Cl

IT 592-51-8, 4-Pentenitrile 16545-78-1,
 cis-3-Pentenitrile 20068-02-4, cis-2-Methyl-2-butenenitrile
 25899-50-7, cis-2-Pentenitrile 26294-98-4,
 trans-2-Pentenitrile 30574-97-1, trans-2-Methyl-2-butenenitrile
 RL: NUU (Other use, unclassified); USES (Uses)
 (method of in-process recycling of Lewis acids in production of nitriles by hydrocyanation)
 RN 592-51-8 HCAPLUS
 CN 4-Pentenitrile (CA INDEX NAME)

H₂C=CH-CH₂-CH₂-CN

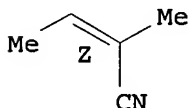
RN 16545-78-1 HCAPLUS
 CN 3-Pentenitrile, (3Z)- (9CI) (CA INDEX NAME)

Double bond geometry as shown.



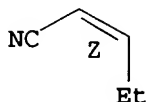
RN 20068-02-4 HCAPLUS
 CN 2-Butenenitrile, 2-methyl-, (2Z)- (CA INDEX NAME)

Double bond geometry as shown.



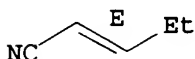
RN 25899-50-7 HCAPLUS
 CN 2-Pentenitrile, (2Z)- (CA INDEX NAME)

Double bond geometry as shown.



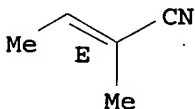
RN 26294-98-4 HCAPLUS
CN 2-Pentenitrile, (2E)- (CA INDEX NAME)

Double bond geometry as shown.



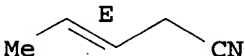
RN 30574-97-1 HCAPLUS
CN 2-Butenenitrile, 2-methyl-, (2E)- (CA INDEX NAME)

Double bond geometry as shown.



IT 16529-66-1, trans-3-Pentenitrile
RL: NUU (Other use, unclassified); RCT (Reactant); RACT (Reactant or reagent); USES (Uses)
(method of in-process recycling of Lewis acids in production of nitriles by hydrocyanation)
RN 16529-66-1 HCAPLUS
CN 3-Pentenitrile, (3E)- (CA INDEX NAME)

Double bond geometry as shown.



RE.CNT 5 THERE ARE 5 CITED REFERENCES AVAILABLE FOR THIS RECORD
ALL CITATIONS AVAILABLE IN THE RE FORMAT

L33 ANSWER 3 OF 14 HCAPLUS COPYRIGHT 2007 ACS on STN
AN 2004:57288 HCAPLUS
DN 140:79246
TI Removal of water from a mixture of water and zinc chloride
IN Jungkamp, Tim; Scheidel, Jens; Luyken, Hermann; Bartsch, Michael; Baumann, Robert; Haderlein, Gerd
PA BASF A.-G., Germany
SO Ger. Offen., 6 pp.
CODEN: GWXXBX
DT Patent
LA German
FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	DE 10231296	A1	20040122	DE 2002-10231296	20020710
	CA 2491791	A1	20040122	CA 2003-2491791	20030704

KATHLEEN FULLER EIC1700 571/272-2505

WO 2004007371	A1	20040122	WO 2003-EP7149	20030704
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW				
RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG				
AU 2003244640	A1	20040202	AU 2003-244640	20030704
EP 1521722	A1	20050413	EP 2003-738116	20030704
EP 1521722	B1	20060405		
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR, BG, CZ, EE, HU, SK				
BR 2003011998	A	20050426	BR 2003-11998	20030704
CN 1668533	A	20050914	CN 2003-816294	20030704
JP 2005538014	T	20051215	JP 2004-520478	20030704
AT 322464	T	20060415	AT 2003-738116	20030704
ES 2261949	T3	20061116	ES 2003-3738116	20030704
MX 2004PA12177	A	20050224	MX 2004-PA12177	20041206
US 2005207968	A1	20050922	US 2004-519513	20041223
IN 2005CN00162	A	20070330	IN 2005-CN162	20050210
PRAI DE 2002-10231296	A	20020710		
WO 2003-EP7149	W	20030704		

AB Water is removed from a mixture of water and zinc chloride by adding an aprotic, polar solvent to the mixture which has a b.p. above the b.p. of water and which is liquid at the b.p. of water, or which forms an azeotrope or hetero-azeotrope with water at the pressure and temperature conditions of the subsequent distillation. The solvent is an aliphatic nitrile, such as cis-2-pentenitrile, trans-2-pentenitrile, cis-3-pentenitrile, trans-3-pentenitrile, 4-pentenitrile, trans-2-methyl-2-butenitrile, cis-2-methyl-2-butenitrile, or 3-methyl-2-butenitrile. HCl is added to the water/ZnCl₂ mixture

IC ICM C01G009-04

ICS B01D003-36

CC 49-5 (Industrial Inorganic Chemicals)

ST water zinc chloride dewatering

azeotropic distn org nitrile

IT Distillation

(azeotropic; removal of water from mixture of water and zinc chloride)

IT Azeotropes

(removal of water from mixture of water and zinc chloride)

IT Nitriles, uses

RL: NUU (Other use, unclassified); USES (Uses)

(removal of water from mixture of water and zinc chloride)

IT 592-51-8, 4-Pentenitrile 4786-24-7, 3-Methyl-2-butenitrile 16529-66-1, trans-3-Pentenitrile 16545-78-1, cis-3-Pentenitrile 20068-02-4, cis-2-Methyl-2-butenitrile 25899-50-7, cis-2-Pentenitrile 26294-98-4, trans-2-Pentenitrile 30574-97-1, trans-2-Methyl-2-

butenenitrile

RL: NUU (Other use, unclassified); USES (Uses)
(removal of water from mixture of water and zinc chloride)

IT 7646-85-7P, Zinc chloride, preparation

RL: PEP (Physical, engineering or chemical process); PUR (Purification or recovery); PYP (Physical process); PREP (Preparation); PROC (Process)
(removal of water from mixture of water and zinc chloride)

IT 7732-18-5, Water, processes

RL: PEP (Physical, engineering or chemical process); PYP (Physical process); REM (Removal or disposal); PROC (Process)
(removal of water from mixture of water and zinc chloride)

IT 7647-01-0, Hydrochloric acid, reactions

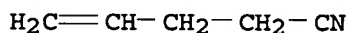
RL: RCT (Reactant); RACT (Reactant or reagent)
(removal of water from mixture of water and zinc chloride)

IT 592-51-8, 4-Pentenitrile 4786-24-7,
3-Methyl-2-butenitrile 16529-66-1, trans-3-Pentenitrile 16545-78-1, cis-3-Pentenitrile 20068-02-4, cis-2-Methyl-2-butenitrile 25899-50-7, cis-2-Pentenitrile 26294-98-4, trans-2-Pentenitrile 30574-97-1, trans-2-Methyl-2-butenitrile

RL: NUU (Other use, unclassified); USES (Uses)
(removal of water from mixture of water and zinc chloride)

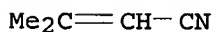
RN 592-51-8 HCAPLUS

CN 4-Pentenitrile (CA INDEX NAME)



RN 4786-24-7 HCAPLUS

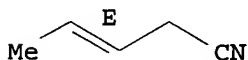
CN 2-Butenenitrile, 3-methyl- (CA INDEX NAME)



RN 16529-66-1 HCAPLUS

CN 3-Pentenitrile, (3E)- (CA INDEX NAME)

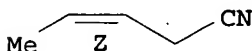
Double bond geometry as shown.



RN 16545-78-1 HCAPLUS

CN 3-Pentenitrile, (3Z)- (9CI) (CA INDEX NAME)

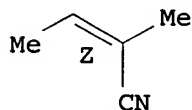
Double bond geometry as shown.



RN 20068-02-4 HCAPLUS

CN 2-Butenenitrile, 2-methyl-, (2Z)- (CA INDEX NAME)

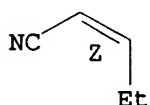
Double bond geometry as shown.



RN 25899-50-7 HCAPLUS

CN 2-Pentenitrile, (2Z)- (CA INDEX NAME)

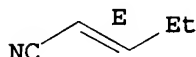
Double bond geometry as shown.



RN 26294-98-4 HCAPLUS

CN 2-Pentenitrile, (2E)- (CA INDEX NAME)

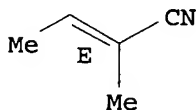
Double bond geometry as shown.



RN 30574-97-1 HCAPLUS

CN 2-Butenenitrile, 2-methyl-, (2E)- (CA INDEX NAME)

Double bond geometry as shown.



IT 7646-85-7P, Zinc chloride, preparation

RL: PEP (Physical, engineering or chemical process); PUR (Purification or recovery); PYP (Physical process); PREP (Preparation); PROC (Process)
(removal of water from mixture of water and zinc chloride)

RN 7646-85-7 HCAPLUS

CN Zinc chloride (ZnCl₂) (CA INDEX NAME)

Cl-Zn-Cl

IT 7732-18-5, Water, processes

RL: PEP (Physical, engineering or chemical process); PYP (Physical process); REM (Removal or disposal); PROC (Process)
(removal of water from mixture of water and zinc chloride)

RN 7732-18-5 HCAPLUS

CN Water (CA INDEX NAME)

H₂O

L33 ANSWER 4 OF 14 HCAPLUS COPYRIGHT 2007 ACS on STN

AN 1997:297495 HCAPLUS

DN 126:277207

TI Process and water-soluble catalysts for the preparation of nitriles by the hydrocyanation of alkenes and cyanoalkenes

IN Huser, Marc; Perron, Robert

PA Rhone-Poulenc Fiber and Resin Intermediates, Fr.; Huser, Marc; Perron, Robert

SO PCT Int. Appl., 33 pp.

CODEN: PIXXD2

DT Patent

LA French

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	WO 9712857	A1	19970410	WO 1996-FR1509	19960927
	W: BR, CA, CN, JP, KR, MX, RU, SG, US				
	RW: AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE				
	FR 2739378	A1	19970404	FR 1995-11689	19950929
	FR 2739378	B1	19971031		
	CA 2231027	A1	19970410	CA 1996-2231027	19960927
	EP 854858	A1	19980729	EP 1996-932661	19960927
	EP 854858	B1	20010613		
	R: BE, DE, FR, GB, IT, NL				
	CN 1198151	A	19981104	CN 1996-197298	19960927
	JP 11501660	T	19990209	JP 1997-514018	19960927
	JP 3739404	B2	20060125		
	BR 9610819	A	19991221	BR 1996-10819	19960927
	RU 2186058	C2	20020727	RU 1998-107641	19960927
	US 5856555	A	19990105	US 1997-832689	19970411
PRAI	FR 1995-11689	A	19950929		
	WO 1996-FR1509	W	19960927		

OS MARPAT 126:277207

AB Alkenes and cyanoalkenes (e.g., 3-pentene nitrile) are hydrocyanated with HCN into nitriles (e.g., adiponitrile) in an aqueous solution of a catalyst that

includes at least one transition metal compound [e.g., nickel bis(cyclopentadiene)], at least one water-soluble monodentate or bidentate phosphine [e.g., sodium 1,2-bis[di(sulfonatophenyl)phosphinomethyl]cyclobutane] ligand, and, optionally, a Lewis acid cocatalyst (e.g., ZnCl₂).

IC ICM C07C253-10

ICS C07C255-04; B01J031-24; C07F009-50

CC 23-19 (Aliphatic Compounds)

Section cross-reference(s): 35, 45, 67

ST adiponitrile prepn cyanopentene hydrocyanation; alkene hydrocyanation prepn nitrile; catalyst water sol alkene hydrocyanation

IT Lewis acids

RL: CAT (Catalyst use); USES (Uses)

(catalysts with transition metal compds. and water-soluble phosphine ligands for the hydrocyanation conversion of alkenes and cyanoalkenes in the aqueous phase into nitriles)

IT Nitriles, preparation

- RL: IMF (Industrial manufacture); SPN (Synthetic preparation); PREP (Preparation)
(dinitriles; process and water-soluble catalysts for the preparation of nitriles by the hydrocyanation of alkenes and cyanoalkenes)
- IT Hydrocyanation
(of alkenes and cyanoalkenes in the aqueous phase in the preparation of nitriles)
- IT Alkenes, reactions
RL: RCT (Reactant); RACT (Reactant or reagent)
(process and water-soluble catalysts for the preparation of nitriles by the hydrocyanation of)
- IT Nitriles, preparation
RL: IMF (Industrial manufacture); SPN (Synthetic preparation); PREP (Preparation)
(process and water-soluble catalysts for the preparation of nitriles by the hydrocyanation of alkenes and cyanoalkenes)
- IT Hydrocyanation catalysts
(transition metal compds. and water-soluble phosphine ligands and Lewis acids for the conversion of alkenes and cyanoalkenes in the aqueous phase into nitriles)
- IT 815-85-0, Stannous tartrate, uses 1293-78-3 7429-91-6D, Dysprosium, bromides or chlorides, uses 7439-91-0D, Lanthanum, bromides or chlorides, uses 7439-94-3D, Lutetium, bromides or chlorides, uses 7440-00-8D, Neodymium, bromides or chlorides, uses 7440-10-0D, Praseodymium, bromides or chlorides, uses 7440-19-9D, Samarium, bromides or chlorides, uses 7440-27-9D, Terbium, bromides or chlorides, uses 7440-30-4D, Thulium, bromides or chlorides, uses 7440-45-1D, Cerium, bromides or chlorides, uses 7440-52-0D, Erbium, bromides or chlorides, uses 7440-53-1D, Europium, bromides or chlorides, uses 7440-54-2D, Gadolinium, bromides or chlorides, uses 7440-60-0D, Holmium, bromides or chlorides, uses 7440-64-4D, Ytterbium, bromides or chlorides, uses 7488-55-3, Stannous sulfate 7646-79-9, Cobalt chloride, uses 7646-85-7, Zinc chloride, uses 7699-45-8, Zinc bromide 7758-94-3, Ferrous chloride 7772-99-8, Stannous chloride, uses 7789-42-6, Cadmium bromide 10031-24-0, Stannous bromide 10108-64-2, Cadmium chloride 10139-47-6, Zinc iodide 10361-92-9, Yttrium chloride 11132-78-8, Manganese chloride 12266-58-9, Nickel bis(acrylonitrile) 12738-03-3, Manganese bromide 14220-17-8, Potassium tetracyanonickelate
RL: CAT (Catalyst use); USES (Uses)
(process and water-soluble catalysts for the preparation of nitriles by the hydrocyanation of alkenes and cyanoalkenes)
- IT 111-69-3P, Adiponitrile
RL: IMF (Industrial manufacture); SPN (Synthetic preparation); PREP (Preparation)
(process and water-soluble catalysts for the preparation of nitriles by the hydrocyanation of alkenes and cyanoalkenes)
- IT 74-90-8, Hydrogen cyanide, reactions 78-79-5, Isoprene, reactions 100-42-5, Styrene, reactions 106-99-0, 1,3-Butadiene, reactions 110-83-8, Cyclohexene, reactions 111-78-4, 1,5-Cyclooctadiene 592-42-7, 1,5-Hexadiene 592-51-8, 4-Pentene nitrile 1319-73-9, Methylstyrene 1335-86-0, Methylcyclohexene 4403-61-6, 2-Methyl-2-butene nitrile 4635-87-4, 3-Pentene nitrile 13284-42-9, 2-Pentene nitrile 16529-56-9, 2-Methyl-3-butene nitrile 26588-32-9, Vinyl naphthalene
RL: RCT (Reactant); RACT (Reactant or reagent)
(process and water-soluble catalysts for the preparation of nitriles by the hydrocyanation of alkenes and cyanoalkenes)
- IT 7646-85-7, Zinc chloride, uses
RL: CAT (Catalyst use); USES (Uses)

(process and water-soluble catalysts for the preparation of nitriles
by the hydrocyanation of alkenes and cyanoalkenes)

RN 7646-85-7 HCAPLUS

CN Zinc chloride (ZnCl₂) (CA INDEX NAME)

Cl-Zn-Cl

IT 592-51-8, 4-Pentene nitrile

RL: RCT (Reactant); RACT (Reactant or reagent)

(process and water-soluble catalysts for the preparation of nitriles
by the hydrocyanation of alkenes and cyanoalkenes)

RN 592-51-8 HCAPLUS

CN 4-Pentenitrile (CA INDEX NAME)

 $\text{H}_2\text{C}=\text{CH}-\text{CH}_2-\text{CH}_2-\text{CN}$

L33 ANSWER 5 OF 14 HCAPLUS COPYRIGHT 2007 ACS on STN

AN 1993:133091 HCAPLUS

DN 118:133091

TI Removal of metal halide catalyst

IN Morita, Haruo; Inagi, Toshiaki; Goto, Takaaki

PA Yokkaichi Chemical Co., Ltd., Japan

SO Jpn. Kokai Tokkyo Koho, 6 pp.

CODEN: JKXXAF

DT Patent

LA Japanese

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	JP 04200643	A	19920721	JP 1990-329872	19901130
PRAI	JP 1990-329872		19901130		

AB The title method involves neutralizing a reaction product containing a metal halide catalyst with an alkali metal phosphate aqueous solution, removing water, and filtering the produced neural salt. Specifically, the reaction product may contain an N-substituted aromatic amine. The catalyst is useful for cyanoethylation or hydroxyalkylation.

IC ICM B01J038-64

ICS B01J038-00; C07C213-04; C07C215-16; C07C253-30; C07C255-24

ICA C07B061-00

CC 67-1 (Catalysis, Reaction Kinetics, and Inorganic Reaction Mechanisms)

Section cross-reference(s): 25

ST metal halide catalyst arom amine; cyanoethylation catalyst metal halide;
hydroxyalkylation catalyst metal halide

IT Catalysts and Catalysis

(metal chloride, removal of)

IT Cyanoethylation catalysts

Hydroxyalkylation catalysts

(metal chlorides, removal of, by neutralizing with alkali metal phosphate)

IT 7446-70-0, Aluminum chloride, uses 7447-39-4, Cupric chloride, uses
7550-45-0, Titanium chloride, uses 7646-78-8, Tin chloride (SnCl₄), uses
7646-85-7, Zinc chloride, uses 7699-45-8,
Zinc bromide 7705-08-0, Ferric chloride, uses 7758-94-3, Ferrous
chloride 7772-99-8, Tin chloride (SnCl₂), uses 7783-49-5, Zinc

fluoride 10139-47-6, Zinc iodide
 RL: CAT (Catalyst use); USES (Uses)
 (catalysts, removal of, by neutralizing with alkali metal phosphate)
 IT 7601-54-9, Sodium phosphate (Na₃PO₄)
 RL: USES (Uses)
 (chloride catalyst removal using)
 IT 75-21-8, Ethylene oxide, reactions
 RL: RCT (Reactant); RACT (Reactant or reagent)
 (reaction of, with Et toluidine, removal of catalysts for)
 IT 62-53-3, Aniline, reactions
 RL: RCT (Reactant); RACT (Reactant or reagent)
 (reaction of, with acrylonitrile, removal of catalysts for)
 IT 107-13-1, Acrylonitrile, reactions
 RL: RCT (Reactant); RACT (Reactant or reagent)
 (reaction of, with aniline, removal of catalysts for)
 IT 102-27-2
 RL: RCT (Reactant); RACT (Reactant or reagent)
 (reaction of, with ethylene oxide, removal of catalysts for)
 IT 7646-85-7, Zinc chloride, uses
 RL: CAT (Catalyst use); USES (Uses)
 (catalysts, removal of, by neutralizing with alkali metal phosphate)
 RN 7646-85-7 HCAPLUS
 CN Zinc chloride (ZnCl₂) (CA INDEX NAME)

Cl-Zn-Cl

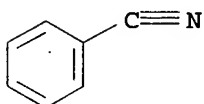
IT 107-13-1, Acrylonitrile, reactions
 RL: RCT (Reactant); RACT (Reactant or reagent)
 (reaction of, with aniline, removal of catalysts for)
 RN 107-13-1 HCAPLUS
 CN 2-Propenenitrile (CA INDEX NAME)

H₂C=CH-C≡N

L33 ANSWER 6 OF 14 HCAPLUS COPYRIGHT 2007 ACS on STN
 AN 1986:446042 HCAPLUS
 DN 105:46042
 TI Reactivity of some organic compounds with supercritical water
 AU Houser, Thomas J.; Tiffany, David M.; Li, Zhuangjie; McCarville, Michael E.; Houghton, Michael E.
 CS Dep. Chem., West. Michigan Univ., Kalamazoo, MI, 49008, USA
 SO Fuel (1986), 65(6), 827-32
 CODEN: FUELAC; ISSN: 0016-2361
 DT Journal
 LA English
 AB This study was initiated to determine some of the chemical reactions that occur during the supercrit. fluid extraction of coal, using model compds. to simulate mol. structures found in coal. Water was chosen as the fluid because of its unique chemical and phys. properties at critical conditions.
 Two primary functions of coal processing are the removal of hetero atoms and the depolymn. of larger mols.; thus the reactions of quinoline [91-22-5] and isoquinoline [119-65-3] were extensively examined, with very brief studies made of benzonitrile [100-47-0], aniline [62-53-3], Tetralin [119-64-2], dihydroanthracene [613-31-0] and ethylbenzene

[100-41-4]. In addition, since ZnCl_2 was used as a hydrogenation catalyst and in the hydrocracking of aroms., it was added in some expts. to increase the reactivity of some of the compds. The compds. studied were more reactive and reacted by different mechanisms in the presence of supercrit. water than when undergoing inert pyrolysis. The product distributions from the 2 quinolines indicated that they reacted by very different mechanisms; possible reaction schemes for these are discussed. In addition, .apprx.70% of the N from the consumed quinolines and aniline was removed in the water layer as NH_3 , alkyl side chains on aroms. were somewhat reactive, and some C atoms are oxidized by the water, thus providing a source of H for the formation of other products.

CC 51-21 (Fossil Fuels, Derivatives, and Related Products)
 ST coal liquefaction supercrit water reaction; org compd reaction supercrit water
 IT Coal liquefaction
 (solvent extraction, supercrit. water reaction with organic compds. in relation to)
 IT 62-53-3, reactions 91-22-5, reactions 100-41-4, reactions 100-47-0, reactions 119-64-2 119-65-3 613-31-0
 RL: RCT (Reactant); RACT (Reactant or reagent)
 (reaction of, with supercrit. water, coal extraction in relation to)
 IT 7732-18-5, reactions
 RL: RCT (Reactant); RACT (Reactant or reagent)
 (supercrit., reaction of, with organic compds., coal extraction in relation to)
 IT 100-47-0, reactions
 RL: RCT (Reactant); RACT (Reactant or reagent)
 (reaction of, with supercrit. water, coal extraction in relation to)
 RN 100-47-0 HCAPLUS
 CN Benzonitrile (CA INDEX NAME)



IT 7732-18-5, reactions
 RL: RCT (Reactant); RACT (Reactant or reagent)
 (supercrit., reaction of, with organic compds., coal extraction in relation to)
 RN 7732-18-5 HCAPLUS
 CN Water (CA INDEX NAME)

H_2O

L33 ANSWER 7 OF 14 HCAPLUS COPYRIGHT 2007 ACS on STN
 AN 1982:168168 HCAPLUS
 DN 96:168168
 TI Heavy metal ion absorbent
 PA Toyobo Co., Ltd., Japan
 SO Jpn. Kokai Tokkyo Koho, 4 pp.
 CODEN: JKXXAF

KATHLEEN FULLER EIC1700 571/272-2505

DT Patent
 LA Japanese
 FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	JP 57004229	A	19820109	JP 1980-78540	19800610
	JP 63023829	B	19880518		
PRAI	JP 1980-78540	A	19800610		
AB	Cr6+ and UO22+ ions are removed from water by protein/acrylonitrile [107-13-1] fibers. Thus, a mixture of milk casein 2.2 and acrylonitrile 5 (in 43 parts 60% aqueous ZnCl2) was stirred at 20° with (NH4)2S2O8 0.06 parts for 2 h, defoamed, and spun through a die into 30% aqueous ZnCl2 at 0°. The resulting fiber was water-washed, stretched 7-fold in steam at 120° to 2 denier, shrunk by 16%, dried, and coiled. Such fibers 1 g were stirred for 1 h with an aqueous solution 100 mL containing Cr6+, 1, 5, 10 and 50 ppm and removed 100, 98, 98, and 92%, resp. The process is useful for industrial wastewater treatment and U recovery from seawater.				
IC	B01J020-26; B01D015-00; C02F001-28				
CC	60-3 (Waste Treatment and Disposal) Section cross-reference(s): 54				
ST	chromium removal wastewater acrylic fiber; acrylonitrile protein fiber chromium wastewater; uranyl adsorption seawater acrylic fiber; adsorption fiber metal wastewater treatment				
IT	Caseins, compounds RL: PROC (Process) (acrylonitrile-grafted, fibers, wastewater treatment with, hexavalent chromium removal by)				
IT	Waters, ocean (uranyl recovery from, by acrylonitrile-casein polymer fiber adsorption)				
IT	Acrylic fibers, uses and miscellaneous RL: USES (Uses) (acrylonitrile-casein polymers, wastewater treatment with, hexavalent chromium removal by)				
IT	Wastewater treatment (adsorption, heavy metal removal in, by acrylonitrile-casein polymer fibers)				
IT	Metals, uses and miscellaneous RL: REM (Removal or disposal); PROC (Process) (heavy, removal of, from wastewaters, by acrylonitrile-casein polymer fiber absorption)				
IT	107-13-1D, polymers with casein RL: PROC (Process) (fibers of, wastewater treatment with, hexavalent chromium removal by)				
IT	16637-16-4P RL: PREP (Preparation) (recovery of, from seawater, by acrylonitrile-casein polymer fiber adsorption)				
IT	7440-47-3, uses and miscellaneous RL: USES (Uses) (removal of hexavalent, from wastewaters, by acrylonitrile-casein polymer fiber adsorption)				
IT	107-13-1D, polymers with casein RL: PROC (Process) (fibers of, wastewater treatment with, hexavalent chromium removal by)				
RN	107-13-1 HCAPLUS				
CN	2-Propenenitrile (CA INDEX NAME)				



L33 ANSWER 8 OF 14 HCAPLUS COPYRIGHT 2007 ACS on STN

AN 1975:482221 HCAPLUS

DN 83:82221

TI Zinc chloride-ammonium chloride double salts from
zinc chloride and/or ammonia and/or hydrogen
chloride-containing waste gases

IN Turkolmez, Sedat

PA Switz.

SO Ger. Offen., 9 pp.

CODEN: GWXXBX

DT Patent

LA German

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	DE 2452409	A1	19750515	DE 1974-2452409	19741105
	CH 599051	A5	19780512	CH 1973-15621	19731107
PRAI	CH 1973-15621	A	19731107		

AB ZnCl_2 , NH_3 , and/or HCl in waste gases evolved from hot galvanizing are removed by scrubbing with water or dilute HCl . The scrubbing solution is analyzed and the amount is added of ZnO , HCl , ZnCl_2 , and/or NH_3 needed to adjust the composition to that of the double salt ($\text{ZnCl}_2 \cdot \text{NH}_4\text{Cl}$, $\text{ZnCl}_2 \cdot 2\text{NH}_4\text{Cl}$, or $\text{ZnCl}_2 \cdot 3\text{NH}_4\text{Cl}$) used in the galvanizing flux. The Fe^{2+} is oxidized with air, H_2O_2 , or Cl to Fe^{3+} . The double salt solution or crystals after concentration are returned to the galvanizing process.

IC C01G; C23C

CC 49-6 (Industrial Inorganic Chemicals)

Section cross-reference(s): 55, 59, 60

ST ammonium zinc chloride crystn; galvanizing bath fume
recovery

IT Waste gases

(from galvanizing bath, waste water from scrubbing of,
ammonium zinc chloride recovery from)

IT Galvanization

(waste water from scrubbing gases from, ammonium zinc
chloride double salt crystallization from)

IT 14639-97-5P 14639-98-6P 15203-88-0P

RL: PREP (Preparation)

(recovery of, from scrubbing waste gas of galvanizing bath)

L33 ANSWER 9 OF 14 HCAPLUS COPYRIGHT 2007 ACS on STN

AN 1965:79181 HCAPLUS

DN 62:79181

OREF 62:14024d-f

TI Producing heat-resistant semiconductor materials from nitriles

IN Kargin, V. A.; Kabanov, V. A.

SO 1 p.

DT Patent

LA Unavailable

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	US 3164555		19650105	US 1961-90904	19610216
PRAI	US		19610216		

AB Heat-resistant semiconductor materials are produced by the polymerization of fatty and aromatic nitriles, e.g. acetonitrile, benzonitrile, and propionitrile, using the chlorides of the metals of Groups II, III, IV, V, and VIII as catalysts. For example, 20 g. dried and purified acetonitrile was placed in an autoclave and 6 g. ZnCl_2 added. The autoclave is purged with dry N, sealed, heated to 200° and held 8 hrs. A brown-black polymer is formed which is treated with water to remove ZnCl_2 . The washed polymer is soluble in H_2SO_4 , H_3PO_4 , and HCOOH . The sp. conductivity of the dried powder at room temperature is 10^{-6}

mho and it withstands heating to 500° without noticeable decomposition. The same reaction conducted 8 hrs. at 250° yields a polymer insol. in organic and inorg. solvents. This polymer withstands heating to $500-600^\circ$ and has a sp. conductance of 10^{-5} mho.

INCL 260002000

CC 9 (Electric and Magnetic Phenomena)

IT Nitriles

(elec. semiconducting heat-resistant)

IT Catalysts and Catalysis

(in polymerization, of nitriles)

IT Semiconductors, electric

(nitrile polymers)

IT Conductivity, electric and(or) Conduction, electric

(of nitrile polymers)

IT Polymerization

(of nitriles, to heat-resistant semiconductors)

IT Propionitrile, homopolymer

(heat-resistant semiconducting)

IT 7446-70-0, Aluminum chloride

(catalysts in polymerization, of nitriles)

IT 10025-91-9, Antimony chloride, SbCl_3

(catalysts, in polymerization of nitriles)

IT 100-47-0, Benzonitrile, polymers of 26809-02-9, Acetonitrile, homopolymer

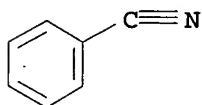
(heat-resistant semiconducting)

IT 100-47-0, Benzonitrile, polymers of

(heat-resistant semiconducting)

RN 100-47-0 HCAPLUS

CN Benzonitrile (CA INDEX NAME)



L33 ANSWER 10 OF 14 HCAPLUS COPYRIGHT 2007 ACS on STN

AN 1964:61406 HCAPLUS

DN 60:61406

OREF 60:10822e-g

TI Copolymerization with monomer complexes

PA Esso Research and Engineering Co.

SO 6 pp.

DT Patent

LA Unavailable

FAN.CNT 1

PATENT NO.

KIND

DATE

APPLICATION NO.

DATE

KATHLEEN FULLER EIC1700 571/272-2505

PI GB 946052 19640108 GB 1962-3005 19620126
 PRAI US 19610301

AB A monomer containing neg. groups that do not promote electron-pair release at the double bond and that responds to free-radical initiation is complexed with a Friedel-Crafts catalyst, and the complexed monomer is copolymd. with a 2nd monomer that responds to Friedel-Crafts copolymn. The 1st monomer is a polar vinyl or vinylidene compound, and the 2nd is an olefin or substituted olefin. In an example, acrylonitrile (I) (2 mol) and 2 mol 2-methyl-1-pentene, containing hydroperoxides, under N, was stirred and 2 mol 98% ZnCl₂ added at room temperature, then heated to 63° and stirred for 3 h. upon which polymer formed. The monomer was decanted, and after ZnCl₂ was removed by repeated treatment with H₂O, the polymer was twice dissolved in acetone and precipitated by H₂O, then dried at 60° for 16 h. at 180 mm. to give a 51% yield, based on I, of a product containing C 75.50, H 9.80, and N 13.39%; it formed fibers and cast films. Methacrylonitrile was similarly copolymd., also methallyl chloride with I. No resin yield was obtained on omission of ZnCl₂; the yield dropped to 8.5% when peroxide was omitted. Propylene (0.2 mol) and 0.6 mol I were copolymd. in a bomb in the presence of 0.2 mol ZnCl₂, 2 drops cumene hydroperoxide, and 0.2 mol acetone at 100° for 1 h. at 295 lb./in.² gage, filtered, the polymer precipitated with water, and solvents removed under low vacuum to yield a copolymer containing 54.4% I. Isobutylene and I were similarly copolymd. When acrylic acid and 1-hexene were copolymd. likewise at atmospheric pressure in the presence of Et₂O, some acetone-sol, product was also obtained containing 20.75% O.

IC C08F

CC 45 (Synthetic High Polymers)

IT Hydroperoxides
 (catalysts from Friedel-Crafts complexes of vinyl or vinylidene compds. and, in polymerization of olefins with vinyl or vinylidene compds.)

IT Catalysts and Catalysis
 (in polymerization of olefins, with vinyl or vinylidene compds., Friedel-Crafts compds. with vinyl or vinylidene compds. as)

IT Polymerization
 (of olefins, with vinyl or vinylidene compds., Friedel-Crafts complexes of vinyl or vinylidene compds. as catalysts in)

IT Vinyl compounds
 (polymerization of, with olefins, Friedel-Crafts complexes of vinyl compds. as catalysts in)

IT Olefins
 (polymerization of, with vinyl or vinylidene compds., Friedel-Crafts complexes of vinyl or vinylidene compds. as catalysts in)

IT 1-Pentene, 2-methyl-, polymer with acrylonitrile
 RL: PREP (Preparation)

IT Zinc, with vinyl or vinylidene compds.
 (catalysts from free-radical-forming compds. and, in polymerization of olefins with vinyl or vinylidene compds.)

IT 1-Hexene, polymer with 2-vinyl-6,6-dimethylnorpinane
 (with vinyl or vinylidene compds., Friedel-Crafts complexes of vinyl or vinylidene compds. as catalysts in)

IT 96-33-3, Acrylic acid, methyl ester
 (catalysts from Friedel-Crafts compds. and, in polymerization of Me acrylate with olefins)

IT 107-02-8, Acrolein
 (catalysts from Friedel-Crafts compds. and, in polymerization of acrolein with

olefins)

IT 78-67-1, Propionitrile, 2,2'-azobis[2-methyl- 94-36-0, Benzoyl
peroxide
(catalysts from Friedel-Crafts complexes of vinyl or vinylidene compds.
and, in polymerization of olefins with vinyl or vinylidene compds.)

IT 96-10-6, Aluminum, chlorodiethyl-
(catalysts from TeCl_2 , TiCl_3 and, in polymerization of olefins)

IT 7440-21-3, Silicon
(catalysts from TiCl_4 and, in polymerization of olefins in presence of Al
compds.)

IT 107-13-1, Acrylonitrile, compound with Friedel-Crafts compds.
(catalysts, in polymerization of acrylonitrile with olefins)

IT 2143-69-3, Vinylidene
(compds., polymerization of, with olefins, Friedel-Crafts complexes of
vinylidene compds. as catalysts in)

IT 126-98-7, Methacrylonitrile
(complexes with Friedel-Crafts compds., catalysts, in polymerization of
methacrylonitrile with olefins, and polymer with methacrylic acid
esters, with olefins, Friedel-Crafts complexes of methacrylonitrile as
catalysts in)

IT 79-10-7, Acrylic acid
(polymerization of (and acrylic acid derivs.), with olefins, Friedel-Crafts
complexes of acrylic acid as catalysts in)

IT 115-07-1, Propene
(polymerization of, vinyl Friedel-Crafts complexes of vinyl or vinylidene
compds. as catalysts in)

IT 107-13-1, Acrylonitrile
(polymerization of, with olefins, Friedel-Crafts compds. with acrylonitrile
as
catalysts in)

IT 115-11-7, Propene, 2-methyl- (isobutylene)
(polymerization of, with vinyl or vinylidene compds., Friedel-Crafts
complexes
of vinyl or vinylidene compds. as catalysts in)

IT 2678-47-9, Acrylic acid, compound with acrylamide (2:1)
(with Friedel-Crafts compds., catalysts, in polymerization of acrylic acid
with olefins)

IT 879631-18-2, Vinyl acetate, compound with PhCN
(with Friedel-Crafts products, catalysts, in polymerization of olefins with
vinyl acetate)

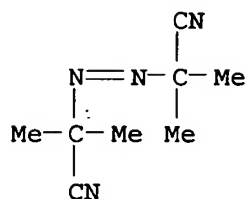
IT 108-05-4, Vinyl acetate
(with olefins, Friedel-Crafts complexes of vinyl acetate as catalysts
in)

IT 7637-07-2, Boron fluoride
(with vinyl or vinylidene compds., catalysts from free-radical-forming
compds. and, in polymerization of olefins with vinyl or vinylidene compds.)

IT 78-67-1, Propionitrile, 2,2'-azobis[2-methyl-
(catalysts from Friedel-Crafts complexes of vinyl or vinylidene compds.
and, in polymerization of olefins with vinyl or vinylidene compds.)

RN 78-67-1 HCAPLUS

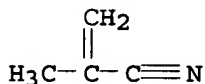
CN Propanenitrile, 2,2'-(1,2-diazenediyl)bis[2-methyl- (CA INDEX NAME)



IT 107-13-1, Acrylonitrile, compound with Friedel-Crafts compds.
(catalysts, in polymerization of acrylonitrile with olefins)
RN 107-13-1 HCAPLUS
CN 2-Propenenitrile (CA INDEX NAME)



IT 126-98-7, Methacrylonitrile
(complexes with Friedel-Crafts compds., catalysts, in polymerization of
methacrylonitrile with olefins, and polymer with methacrylic acid
esters, with olefins, Friedel-Crafts complexes of methacrylonitrile as
catalysts in)
RN 126-98-7 HCAPLUS
CN 2-Propenenitrile, 2-methyl- (CA INDEX NAME)



IT 107-13-1, Acrylonitrile
(polymerization of, with olefins, Friedel-Crafts compds. with acrylonitrile
as
catalysts in)
RN 107-13-1 HCAPLUS
CN 2-Propenenitrile (CA INDEX NAME)



L33 ANSWER 11 OF 14 HCAPLUS COPYRIGHT 2007 ACS on STN
AN 1952:7100 HCAPLUS
DN 46:7100
OREF 46:1242g-i,1243a-f
TI Process for resolving emulsions
IN Kirkpatrick, Willard H.; Wilson, Doyne L.
PA Visco Products Co.
DT Patent
LA Unavailable
FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	US 2568738		19510925	US 1947-741678	19470415
AB	Water-in-petroleum oil emulsions are resolved by agitation with small quantities of the condensation products of modified alkylolamines				

KATHLEEN FULLER EIC1700 571/272-2505

with fatty substances containing 8-32 C atoms. Thus triethanolamine 700 is modified by heating at 230° with $ZnCl_2$ 35 until water 150 parts is removed. Castor oil 630 and modified triethanolamines 300 parts are heated at 160-225° for 12 hrs. with a SO_2 hydrocarbon extract 100 until water 66 parts is removed. The condensed intermediate is diluted with hydrocarbon solvent and isopropanol, and partially neutralized with HCl. The dehydration of the triethanolamine may be carried out simultaneously with the reaction with castor oil. Alkylolamines may also be modified by polymerization without loss of water at elevated temperature, in the presence of acidic metal salts, S, BF_3 , etc. In U.S. 2,568,739, Willard H. Kirkpatrick and Earl T. Kocher (to same assignee), water-wettable modified alkyl resins are prepared by causing a mixture of a polybasic acid, a mixed ester (I), and an alkylolamine, to react above 165°. I is the esterification product of a polyethylene glycol and a mixture of an unsatd. fatty acid containing 8-32 C atoms and a rosin acid. The alkylolamine modifies the resinous product to prevent the formation of insol., infusible resins. In U.S. 2,568,740, Willard H. Kirkpatrick and Doyne L. Wilson (to same assignee) oil-in-water demulsifying surface-active agents (I) are combined with a hydrophilic colloid (e.g. glue, dextrin), a gel-liquefying agent (urea, dicyanodi-amide, etc.) and water. $CaCl_2$ and $ZnCl_2$ may replace I. Examples of I are: the reaction products of a fatty acid containing at least 8 C atoms or its ester and an alkylolamine, aliphatic polyamine or polymerized alkylolamine. In U.S. 2,568,741, Willard H. Kirkpatrick and Earl T. Kocher (to same assignee), mixed esters of polyethylene glycols containing an aliphatic acid having at least 8 C atoms and a rosin acid are treated with the reaction products of a mixture of blown tall oil, triethanolamine, and $ZnCl_2$. In U.S. 2,568,742, Willard H. Kirkpatrick (to same assignee), demulsifying compns. are prepared by dissolving in water a hydrophilic un-ionized colloid, e.g. glue, starch, or dextrin, and a molecularly dehydrated reaction product (I) of $ZnCl_2$ (3-10%) and triethanolamine. I is preferably partially neutralized with HCl. Urea may be added to inhibit gel formation. In U.S. 2,568,743, tall oil is caused to react with triethanolamine at 250°. Water is removed by distillation and the resulting compound is diluted with a SO_2 extract of petroleum hydrocarbons. In U.S. 2,568,744, Earl T. Kocher (to same assignee), the residue remaining from the reaction of an alkylene oxide and NH_3 after removal of the monoamines is caused to react with $ZnCl_2$ at 200°, whereby water is removed.

The demulsifying compound is then obtained by partially neutralizing with HCl. In U.S. 2,568,745, Willard H. Kirkpatrick (to same assignee), a water solution is prepared which contains the partially neutralized, molecularly dehydrated condensation product of $ZnCl_2$ and the residue (I) remaining from the reaction of an alkylene oxide and NH_3 after removal of the monoamines and a hydrophilic colloid, e.g. glue or dextrin. In addition, urea may be added as a gel-inhibiting material. In the preparation

of I the molar ratio of alkylene oxide to N does not exceed 3:1. In U.S. 2,568,746, Willard H. Kirkpatrick and Doyne L. Wilson (to same assignee), esters and salts of alkenyl succinic acids, in which the alkenyl group contains 5-30 C atoms, are prepared by causing the acids to react with aniline, NH_3 , diethylene triamine, diethanolamine, lime, NaOH, pine oil, allyl alc. or phenol, etc., or mixts. thereof. The materials may be diluted with a SO_2 extract. In U.S. 2,568,747, Willard H. Kirkpatrick and Earl T. Kocher (to same assignee), water-wettable modified alkyd resins are prepared by causing a tall oil (I) ester of a polyethylene glycol, triethanolamine, and a dibasic acid, e.g. phthalic acid, to react. I may be replaced by a mixture of an unsatd. fatty acid containing at least 8 C atoms and a rosin acid. In U.S. 2,568,748, Willard H. Kirkpatrick and Doyne L.

Wilson (to same assignee), alkylolamines are partially dehydrated, caused to react with higher fatty acids, rosin, resin acids or naphthenic acids, or their esters or amides, the reaction product is partially neutralized and admixed with one or more un-ionized hydrophilic colloids, e.g. glue or dextrin, in the presence of a diluent. Urea may be added.

- CC 22 (Petroleum, Lubricants, and Asphalt)
- IT Resinous products
 - (alkyd, from alkylolamines, mixed esters and polybasic acids, for breaking petroleum emulsions)
- IT Resinous products
 - (alkyd, from dibasic acids polyethylene glycol esters, and triethanolamine for breaking petroleum emulsions)
- IT Alcohols
 - (amino, alkyd resins from mixed esters, polybasic acids and, for petroleum demulsification)
- IT Alcohols
 - (amino, condensation products with fatty materials, for petroleum demulsification)
- IT Alcohols
 - (amino, dehydrated by $ZnCl_2$, for petroleum demulsification)
- IT Alcohols
 - (amino, dehydrated by $ZnCl_2$, mixts. with hydrophilic colloids, for petroleum demulsification)
- IT Alcohols
 - (amino, reaction products of dehydrated, with acids, amides or esters, mixts. with hydrophilic colloids for petroleum demulsification)
- IT Alcohols
 - (amino, reaction products of, or their polymers, with fatty acids or their esters, petroleum demulsifiers from)
- IT Emulsions
 - (breaking petroleum, alkyd resins from polyethylene glycol esters, triethanolamine and dibasic acids for)
- IT Emulsions
 - (breaking petroleum, alkylolamine-mixed ester-polybasic acid alkyd resin for)
- IT Emulsions
 - (breaking petroleum, dehydrated amino alcs. for)
- IT Emulsions
 - (breaking petroleum, dehydrated amino-alc. mixts. with hydrophilic colloids for)
- IT Emulsions
 - (breaking petroleum, dehydrated amino-alc. reaction products with acid, amides or esters for)
- IT Emulsions
 - (breaking petroleum, hydrophilic colloid compns. with reaction products of triethanolamine and $ZnCl_2$ for)
- IT Emulsions
 - (breaking petroleum, resins from mixed esters of polyethylene glycols and reaction products of tall oil, triethanolamine and $ZnCl_2$ for)
- IT Emulsions
 - (breaking petroleum, succinic acids and their esters and salts for)
- IT Emulsions
 - (breaking petroleum, surface-active agent mixts. with hydrophilic colloids and gel-liquefying agents for)
- IT Emulsions
 - (breaking petroleum, tall-oil reaction products with triethanolamine for)
- IT Emulsions
 - (breaking petroleum, with condensation products of modified

- alkylolamines with fatty materials)
- IT Fatty materials
(condensation products with modified alkylolamines, for breaking petroleum emulsions)
- IT Castor oil
(condensation with dehydrated triethanolamine, petroleum demulsifiers by)
- IT Petroleum
(emulsions of, alkylolamine-mixed ester-polybasic acid alkyd resins for breaking)
- IT Petroleum
(emulsions of, breaking with condensation products of modified alkylolamines with fatty materials)
- IT Petroleum
(emulsions of, dehydrated amino alc. mixts. with hydrophilic colloids for breaking)
- IT Petroleum
(emulsions of, dehydrated amino alc. reaction products with acids, amides or esters for breaking)
- IT Petroleum
(emulsions of, dehydrated amino alcs. for breaking)
- IT Petroleum
(emulsions of, hydrophilic colloid compns. with reaction products of triethanol-amine and $ZnCl_2$ for breaking)
- IT Petroleum
(emulsions of, resins (alkyd) from polyethylene glycol esters, triethanolamine and dibasic acid for breaking)
- IT Petroleum
(emulsions of, resins from mixed esters of polyethylene glycols and reaction products of tall oil, triethanolamine and $ZnCl_2$ for breaking)
- IT Petroleum
(emulsions of, succinic acids and their ester and salts for breaking)
- IT Petroleum
(emulsions of, surface-active agent mixts. with hydrophilic colloids and gel-liquefying agents for breaking)
- IT Petroleum
(emulsions of, tall-oil reaction products with triethanolamine for breaking)
- IT Tall oil
(esters of, with polyethylene glycols, alkyd resins from dibasic acids, triethanolamine and, for petroleum demulsification)
- IT Resin acids or Rosin acids
(esters, with polyethylene glycols, resins from reaction products of tall oil, triethanolamine and $ZnCl_2$ and, for breaking petroleum emulsions)
- IT Resinous products
(from esters (mixed) of polyethylene glycols and reaction products of tall oil, triethanolamine and $ZnCl_2$, for breaking petroleum emulsions)
- IT Catalysts
(in dehydration, of triethanolamine, $ZnCl_2$ as)
- IT Colloids
Colloids
Colloids
(mixts. of, with dehydrated amino alcs., for breaking petroleum emulsions)
- IT Colloids
(mixts. of, with gel-liquefying agents and surfaceactive agents for breaking petroleum emulsions)

- IT Glue
(mixts. with amino alc. reaction products with acids, amides or esters,
for petroleum demulsification)
- IT Glue
Glue
(mixts. with dehydrated amino alcs., for petroleum demulsification)
- IT Resin acids or Rosin acids
Resin acids or Rosin acids
(mixts. with fatty acids, esters with polyethylene glycols, alkyd
resins from alkylolamines, polybasic acids and, for breaking petroleum
emulsions)
- IT Glue
(mixts. with gel-liquefying agents and $\text{CaCl}_2\text{-ZnCl}_2$ mixture or
surface-active agents, for petroleum demulsification)
- IT Surface-active substances (capillary- or interface-active substances)
(mixts. with gel-liquefying agents and hydrophilic colloids for
breaking petroleum emulsions)
- IT Polymerization
(of amino alcs. in making petroleum demulsifiers)
- IT Condensation, chemical
(of fatty materials with modified alkylolamines)
- IT Esters
Esters
(of glycols (polyethylene), with fatty acid-rosin acid mixts., alkyd
resins from alkylolamines, polybasic acids and, for breaking petroleum
emulsions)
- IT Esters
(of glycols, with carboxy and rosin acids, resins from reaction
products of tall oil, triethanolamine and ZnCl_2 and, for
breaking petroleum emulsions)
- IT Dehydration (chemical)
(of nitrilotriethanol with ZnCl_2 catalysts)
- IT Oils
(pine, esterification of alkenyl succinic acids with, for petroleum
demulsifiers)
- IT Tall oil
(reaction products of blown, with triethanolamine and ZnCl_2 ,
resins from esters of polyethylene glycols and, for petroleum
demulsification)
- IT Amines
(reaction products of poly-, with fatty acids or their esters,
petroleum demulsifiers from)
- IT Sulfonic acids
(reaction products of polycyclic alkylarene, with polymerized
diolefins, and their condensation products with amines, surface-active)
- IT Fatty acids
(reaction products of, with alkylolamines or polyamines, petroleum
emulsifiers from)
- IT Fatty acids
(reaction products of, with amino alcs., mixts. with hydrophilic
colloids for breaking petroleum emulsions)
- IT Tall oil
(reaction products of, with triethanolamine, for petroleum
demulsification)
- IT Esters
(reaction products with alkylolamines or polyamines, petroleum
emulsifiers from)
- IT Amides
Esters
Naphthenic acids

Resin acids or Rosin acids

Rosin

(reaction products with amino alcs., mixts. with hydrophilic colloids for breaking petroleum emulsions)

- IT 110-15-6, Succinic acid
(alkenyl derivs., and their esters and salts, for breaking petroleum emulsions)
- IT 7646-85-7, Zinc chloride
(amino alc. dehydration and modification by, for petroleum demulsifiers)
- IT 7646-85-7, Zinc chloride
(as catalyst, in dehydration of triethanolamine)
- IT 102-71-6, Ethanol, 2,2',2''-nitrilotri-
(dehydration of, with $ZnCl_2$ and condensation of modified amine with castor oil, petroleum demulsifiers by)
- IT 108-95-2, Phenol
(esterification of alkenyl succinic acids with, for petroleum demulsifiers)
- IT 111-42-2, Ethanol, 2,2'-iminodi-
(esters of, with alkenyl succinic acids, petroleum-demulsifying)
- IT 25322-68-3, Polyethylene glycol
(esters with fatty acid-rosin acid mixts., alkyd resins from alkylolamines and polybasic acids, for breaking petroleum emulsions)
- IT 25322-68-3, Polyethylene glycol
(esters, alkyd resins from dibasic acids, triethanolamine and, for breaking petroleum emulsions)
- IT 25322-68-3, Polyethylene glycol
(esters, resins from reaction products of tall oil, triethanolamine and $ZnCl_2$ and, for breaking petroleum emulsions)
- IT 62-53-3, Aniline
(mixed NH_4 -aniline salts of alkenyl succinic acids, petroleum-demulsifying)
- IT 7646-85-7, Zinc chloride
(mixts. with $CaCl_2$, petroleum demulsifying compns. from glue, urea and)
- IT 10043-52-4, Calcium chloride
(mixts. with $ZnCl_2$, petroleum demulsifying agent from glue, urea and)
- IT 57-13-6, Urea
(mixts. with amino alc. reaction products with acids, amides or esters and hydrophilic colloids, for breaking petroleum emulsions)
- IT 9004-53-9, Dextrin
(mixts. with amino alc. reaction products with acids, amides or esters, for breaking petroleum emulsions)
- IT 57-13-6, Urea
(mixts. with dehydrated amino alcs. and hydrophilic colloids, for breaking petroleum emulsions)
- IT 9004-53-9, Dextrin
(mixts. with dehydrated amino alcs., for breaking petroleum emulsions)
- IT 9004-53-9, Dextrin
(mixts. with gel-liquefying agents and $CaCl_2ZnCl_2$ mixture or surface-active agents, for breaking petroleum emulsions)
- IT 461-58-5, Guanidine, cyano-
(mixts. with hydrophilic colloids and $CaCl_2-ZnCl_2$ mixts. or surface-active agents, for breaking petroleum emulsions)
- IT 57-13-6, Urea
(mixts. with hydrophilic colloids and surfaceactive agents on $CaCl_2-ZnCl_2$ mixture for breaking petroleum emulsions)
- IT 9005-25-8, Starch
(mixts. with triethanolamine- $ZnCl_2$ reaction products for breaking petroleum emulsions)

IT 102-71-6, Ethanol, 2,2',2''-nitrilotri-
(reaction products with $ZnCl_2$, mixts. with hydrophilic
colloids for breaking petroleum emulsions)

IT 111-40-0, Diethylenetriamine
(reaction products with succinic acids, petroleum-demulsifier)

IT 102-71-6, Ethanol, 2,2',2''-nitrilotri-
(reaction products with tall oil and $ZnCl_2$, resins from
esters of polyethylene glycols and, for breaking petroleum emulsions)

IT 7646-85-7, Zinc chloride
(reaction products with tall oil and triethanolamine, resins from
esters of polyethylene glycols and, for breaking petroleum emulsions)

IT 102-71-6, Ethanol, 2,2',2''-nitrilotri-
(reaction products with tall oil, for breaking petroleum emulsions)

IT 7646-85-7, Zinc chloride
(reaction products with triethanolamine, for breaking petroleum
emulsions)

IT 107-18-6, Allyl alcohol
(reaction with alkenyl succinic acids for petroleum demulsifiers)

IT 7664-41-7, Ammonia
(reactions of, with alkenyl succinic acids for petroleum demulsifiers)

IT 102-71-6, Ethanol, 2,2',2''-nitrilotri-
(resins (alkyd) from dibasic acids and polyethylene glycol esters for
breaking petroleum emulsions)

IT 7646-85-7, Zinc chloride
(amino alc. dehydration and modification by, for petroleum
demulsifiers)

RN 7646-85-7 HCAPLUS

CN Zinc chloride ($ZnCl_2$) (CA INDEX NAME)

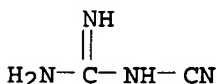
Cl-Zn-Cl

(as catalyst, in dehydration of triethanolamine
(mixts. with $CaCl_2$, petroleum demulsifying compns. from glue, urea and

IT 461-58-5, Guanidine, cyano-
(mixts. with hydrophilic colloids and $CaCl_2$ - $ZnCl_2$ mixts. or
surface-active agents, for breaking petroleum emulsions)

RN 461-58-5 HCAPLUS

CN Guanidine, N-cyano- (CA INDEX NAME)



IT 7646-85-7, Zinc chloride
(reaction products with tall oil and triethanolamine, resins from
esters of polyethylene glycols and, for breaking petroleum emulsions)

RN 7646-85-7 HCAPLUS

CN Zinc chloride ($ZnCl_2$) (CA INDEX NAME)

Cl-Zn-Cl

(reaction products with triethanolamine, for breaking petroleum
emulsions)

L33 ANSWER 12 OF 14 HCAPLUS COPYRIGHT 2007 ACS on STN

AN 1949:4521 HCAPLUS

DN 43:4521

OREF 43:1035d-i,1036a-i,1037a-d

TI Benzils. I. The action of cyanogen on phenols

AU Knobloch, Heinrich; Schraufstatter, Ernst

SO Chemische Berichte (1948), 81, 224-35

CODEN: CHBEAM; ISSN: 0009-2940

DT Journal

LA Unavailable

AB A number of benzils have been prepared by the (CN)₂ synthesis, although some phenols and the naphthols did not react in the desired way. By Karrer and Ferla's method (C.A. 15, 3468) only about 5% resorcil (I), at best, was obtained, and increasing the amount of (CN)₂ or the reaction time did not increase the yield, but the use of condensing agents did. AlCl₃ proved best for this purpose; 200 g. resorcinol in 1.4 l. absolute ether with 200 g. anhydrous AlCl₃, treated 1 h. in the cold with dry HCl gas, then, for about 2 days, simultaneously with (CN)₂ (from 665 g. NaCN in 1280 cc.

water and 1680 g. Cu sulfate in 3340 cc. water, and

dried over P₂O₅) and dry HCl, let stand 3 days, poured into about 1 l. of 10% HCl and 1 kg. crushed ice, and the precipitated diketimide-2HCl (II)

decomposed

by boiling a few min. in 3% HCl, gave 74% I, almost colorless needles from 80% AcOH, m. 263°. The II need not be isolated; if, when the reaction mixture is poured on ice, the HCl concentration is insufficient the product remains in solution in the water layer and, after removal of the ether layer, can be hydrolyzed to I by heating.

The I can also be obtained, more quickly, in 75% yield by cooling the mixture of resorcinol-AlCl₃-ether to about -30° in an autoclave, passing in the (CN)₂ and HCl rapidly, sealing the autoclave, and letting stand 2 days at room temperature. With H₂O₂ and KOH in boiling alc., I gave a little 2,4-(HO)2C₆H₃CO₂H. Derivs. of I: 3,3',5,5'-tetra-Cl (6 g. from 5.5 g. I in cold glacial AcOH with a slow current of Cl), yellow needles from 80% AcOH, m. 220°; 5,5'-di-Br (4.8 g. from 5.5 g. I and 6.6 g. Br in glacial AcOH at room temperature), yellow needles from 80% AcOH, sinters 221°, m. 227°; 3,3',5,5'-tetra-Br (7 g. from 5.5 g. I and

12.6 g. Br), yellow needles from 80% AcOH, m. 259° (decomposition); 5,5'-diiodo (3.2 g. from 2.74 g. I and 5 g. iodine in ice-KOH treated with a little KHSO₃ and poured into an excess of dilute H₂SO₄ and ice), yellow needles from MeOH, m. 205-6°; 5,5'-dinitro (1.5 g. from 2.5 g. I in cold glacial AcOH slowly treated with 2 g. HNO₃ (d. 1.5) and let stand 1 h.), pale yellow needles from 80% AcOH, m. 219°; tetraacetate (70-80% from I refluxed 0.5 h. in excess of Ac₂O), needles from alc. or 80% AcOH, m. 144°; 4,4'-di-Me ether (IIa) (5.4 g. from 12 g. I in 10% NaOH treated dropwise on the water bath with 10.5 cc.

Me₂SO₄), needles from 80% AcOH, m. 141°, gives 2,4-HO(MeO)C₆H₃CO₂H, m. 158°, with H₃O₂, and 1.25 g. IIa with 1.9 g. Br yields 1.1 g.

5,5'-di-Br derivative, [5,2,4-Br(HO)(MeO)C₆H₂CO]₂, yellow needles from benzene, m. 274°. I tetra-Me ether (1.65 g.) and 1.8 g. Br yield 1.3 g.

5,5'-di-Br derivative, pale yellow needles from benzene, m. 259°.

Resorcil 4,4'-di-Et ether (4.8 g. from 13.7 g. I and 14.2 cc. Et₂SO₄ in 10% NaOH heated 1 h. on the water bath), yellow needles from 50%

AcOH, m. 140°; 1.1 g. and 1.2 g. Br give 1 g. 5,5'-di-Br derivative, needles from 80% AcOH, m. 219°.

Tetra-Et ether, from I and excess of Et₂SO₄, needles from 50% alc., m. 155°. The reaction of (CN)₂ with m-MeOC₆H₄OH is complicated by the fact that both the 2- and the 4-MeO compds. may be formed. When 50 g. of the ether was treated as above in ether with AlCl₃, (CN)₂, and HCl, and poured into dilute HCl and ice, the aqueous layer on hydrolysis with boiling HCl yielded an oil which partly solidified on cooling and gave from 50% AcOH 4 g. needles (a); from the

mother liquor water again precipitated an oil which from 50% alc. yielded a little of a crystalline compound (b); from the alc. filtrate water precipitated an oil which, dissolved in NaOH and poured into dilute H₂SO₄, yielded a pale brown powder (c). The ether layer of the original reaction mixture yielded unchanged MeOC₆H₄OH and a crystalline compound (d).

- (a) was the 4,4'-di-Me ether of I, m. 140°; (b) (3% yield), pale yellowish needles from dilute alc., m. 173°, was presumably the 2,2'-di-Me ether; (c) after 2 reprecipitations from Na₂CO₃ with dilute H₂SO₄ m. 90-5° unsharply and did not crystallize (yield, 20%); it gave the tetra-Me ether with Me₂SO₄ and was possibly the 2,4'-di-Me ether; (d) boiled 15 min. in water dissolved and on cooling deposited 4,2-HO(MeO)C₆H₃CO₂H, m. 186° (5%). Apparently, when (CN)₂ adds at the o-position to the MeO group it reacts only to a slight extent with another mol. of the ether. m-EtOC₆H₄OH behaved similarly, giving the Et analogs of (a) and (c) above; it did not form the Et analog of (b), however, but did give 4-hydroxy-2-ethoxybenzoic acid, needles from water, m. 152°, gives no color with FeCl₃ and is converted by Et₂SO₄ into 2,4-(EtO)C₆H₃CO₂H, m. 96°. Unlike m-C₆H₄(OMe)₂, m-C₆H₄(OEt)₂ formed no benzil with (CN)₂, nor did the 4-Cl, 4-Br, or 2-O₂N derivs. of resorcinol or its monoacetate, o- or p-C₆H₄(OH)₂ or their mono- or di-Me ethers, or 1,3,5-C₆H₃(OH)₃. 3,3',5,5'-Tetrabromo-4,4'-dihydroxybenzil (5 g. from 4.9 g. (p-HOC₆H₄CO)₂ and 12.6 g. Br in glacial AcOH), pale yellow needles from glacial AcOH, m. 275°. 2,2',3,3',4,4'-Hexahydroxybenzil (pyrogallil) [45 g. from 125 g. 1,2,3-C₆H₃(OH)₃, ZnCl₂, (CN)₂, and HCl], needles from 80% AcOH, m. 291° (decomposition); the 1,3-di-Me and the tri-Me ether did not react with (CN)₂. 2,2',4,4',5,5'-Isomer (5 g. from 10 g. 1,2,4-C₆H₃(OH)₃), orange-red needles from glacial AcOH, m. 350°; 4,4'-di-Me ether (54% from 2,1,4-MeOC₆H₃(OH)₂), light brown needles from glacial AcOH, m. 263° (decomposition). Phenol, anisole, o-, m-, and p-cresol, thymol, p-BrC₆H₄OH, and p-HOC₆H₄CO₂Me did not react with (CN)₂. 1-Naphthol (25 g.) gave 3 g. yellow needles (III), m. 196° (from 50% AcOH), whose instability made its crystallization and anal.

difficult;

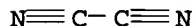
on the basis of its hydrolysis with hot water to 1,2-HOC₁₀H₆CO₂H, m. 186°, and of its dark yellow Br derivative, m. 192° (from dilute AcOH), to 4,1,2-Br(HO)C₁₀H₅CO₂H, m. 235°, III is believed to be the lactone of 1-hydroxy-2-naphthaleneglyoxylic acid. 1,5-C₁₀H₆(OH)₂ gave about 10% red-brown crystals with metallic luster, m. 272°, of probably the lactone of 1,5-dihydroxy-2(or 8)-naphthaleneglyoxylic acid. The above results indicate that the (CN)₂ synthesis gives good yields only with very reactive phenols. Furthermore, if more than 1 nuclear C atom adjacent to the point of attachment of the (CN)₂ is substituted, the (CN)₂ reacts with only 1 mol. of the phenol, apparently because of steric hindrance. Benzils as a rule are yellow, but some noteworthy exceptions were found in this work. A number of the benzils were colorless both in the solid state and in solution, others colorless in the solid form but yellow in solution; [2,4,5-(HO)C₆H₂CO]₂ and [4,2,5-Br(HO)₂C₆H₂CO]₂ are brown-orange. For the detection of benzils, the usual ketone reagents (PhNHNH₂, etc.), o-C₆H₄(NH₂)₂, the Bamberger reaction [Ber. 18, 865(1885)], the Ishidate microchem. test (C.A. 32, 7855.4), and the color reaction with H₂SO₄ often fail or are nonspecific; polarog. detection is more reliable. All the benzils prepared showed characteristic waves. Below are the values for $\pi/2$ (in neg. v.) determined with N NH₄Cl in 75% EtOH and N LiCl in 75% iso-PrOH, resp. (R in (RxC₆H₅-xCO)₂ given): H, 0.59, 0.69; 2-HO, 0.50, 0.57; 5,2-Br(HO), 0.49, -; 2,4-(HO)₂, 0.63, 0.81; 3,5,2,4-Cl₂(HO)₂, 0.42, -; 5,2,4-Br(HO)₂, 0.41, -; 3,5,2,4-Br₂(HO)₂, 0.38, -; 5,2,4-I(HO)₂, 0.50, -; 5,2,4-O₂N(HO)₂, -, 0.25 (0.42, 0.99); 2,4-(AcO)₂, 0.52, -; 2,4-HO(MeO), 0.38, -;

5,2,4-Br(HO)(MeO), 0.31, -; 4,2-HO(MeO) (?), 0.91, -; 2,4-(MeO)₂, 0.78, -;
 5,2,4-Br(MeO)₂, 0.75, -; 2,4-HO(EtO), 0.47, -; 5,2,4-Br(HO)(EtO), 0.45, -;
 2,4-(EtO)₂, 0.83, -; 2,3,4-(OH)₃, 0.46, -; 2,4,5-(HO)₃, 0.76, -;
 4,2,5-MeO(HO)₂, 0.70, -; 4-HO, -, 0.89 (1.23); 3,5,4-Br₂(HO), -, 1.20.

None of the benzils prepared in this work were superior to bromosalicil (IV) in bacteriostatic action against *Staphylococcus aureus*. Whereas IV under the test conditions was still effective at a dilution of 1:320,000, [5,2,4-Br(HO)C₆H₃CO]₂ and [3,5,4-Br₂(HO)C₆H₂CO]₂ were active only at 1:15,000; salicil is more potent (1:20,000) than I (1:10,000) but less powerful than pyrogallil (1:60,000); all the other new benzils showed little or no activity. No definite correlation between structure and bacteriostatic action could be established. Neither p-H₂NC₆H₄CO₂H nor pantothenic acid were antagonistic to the benzils. Addition of 1% serum to the nutritive medium halved the action of IV and 10% completely destroyed its growth-inhibiting activity; this effect of serum is greater on the poly-HO than on the di-HO benzils; pyrogallil is completely inhibited by 1% serum.

- CC 10 (Organic Chemistry)
 IT *Staphylococcus aureus*
 (benzil derivative effect on)
 IT Bactericidal action or Bacteriostatic action
 (of benzil derivs. on *Staphylococcus aureus*)
 IT Phenols
 (reactions with cyanogen)
 IT Gentisil, 4,4'-dibromo-
 RL: PREP (Preparation)
 IT 134-81-6, Benzil
 (and derivs.)
 IT 89-86-1P, β-Resorcylic acid
 RL: PREP (Preparation)
 (formation from 2,2',4,4'-tetrahydroxybenzil)
 IT 2237-36-7P, p-Anisic acid, 2-hydroxy-
 RL: PREP (Preparation)
 (formation from 2,2'-dihydroxyanisil)
 IT 85-26-7P, Salicil 86-48-6P, 2-Naphthoic acid, 1-hydroxy- 523-88-6P,
 Salicil, 5,5'-dibromo- 5394-98-9P, β-Resorcil 5538-39-6P, Benzoic
 acid, 2-ethoxy-4-hydroxy- 5813-37-6P, 2-Naphthoic acid,
 4-bromo-1-hydroxy- 6049-94-1P, Pyrogallil 6706-94-1P, p-Anisil,
 2,2'-dihydroxy- 19074-30-7P, Benzoic acid, 2,4-diethoxy- 82362-01-4P,
 Benzil, 2,2',4,4'-tetramethoxy- 90111-34-5P, o-Anisic acid, 4-hydroxy-
 293750-55-7P, Naphtho[1,2-b]furan-2,3-dione 293750-55-7P,
 2-Naphthaleneglyoxylic acid, 1-hydroxy-, γ-lactone 853787-38-9P,
 Benzil, 2,2',4,4'-tetraethoxy- 853787-39-0P, Benzil,
 3,3',5,5'-tetrabromo-4,4'-dihydroxy- 853787-43-6P, Benzil,
 5,5'-dibromo-2,2',4,4'-tetramethoxy- 853788-47-3P, Benzil,
 2,2',4,4',5,5'-hexahydroxy- 855224-94-1P, Benzil, 2,4'-dihydroxy-2',4'-
 dimethoxy- 855948-08-2P, o-Anisil, 4,4'-dihydroxy- 855948-24-2P,
 p-Anisil, 2,2',5,5'-tetrahydroxy- 857601-30-0P, p-Anisil,
 5,5'-dibromo-2,2'-dihydroxy- 860503-68-0P, β-Resorcil,
 3,3',5,5'-tetrachloro- 860503-70-4P, β-Resorcil, 5,5'-dinitro-
 860503-74-8P, β-Resorcil, 5,5'-diiodo- 860503-95-3P,
 β-Resorcil, 5,5'-dibromo- 860503-96-4P, β-Resorcil,
 tetraacetate 860506-99-6P, Salicil, 4,4'-diethoxy- 860507-01-3P,
 Salicil, 5,5'-dibromo-4,4'-diethoxy- 860714-03-0P, β-Resorcil,
 3,3',5,5'-tetrabromo- 872796-55-9P, 2-Naphthaleneglyoxylic acid,
 1,5-dihydroxy-, γ-lactone 872796-55-9P, Naphtho[1,2-b]furan-2,3-
 dione, 6-hydroxy- 872796-59-3P, 1-Naphthaleneglyoxylic acid,
 4,8-dihydroxy-, δ-lactone 872796-59-3P, Naphtho[1,8-bc]pyran-2,3-
 dione, 6-hydroxy-
 RL: PREP (Preparation)

(preparation of)
 IT 460-19-5, Cyanogen
 (reaction with phenols)
 IT 460-19-5, Cyanogen
 (reaction with phenols)
 RN 460-19-5 HCAPLUS
 CN Ethanedinitrile (CA INDEX NAME)



L33 ANSWER 13 OF 14 HCAPLUS COPYRIGHT 2007 ACS on STN

AN 1908:2681 HCAPLUS

DN 2:2681

OREF 2:637e-i

TI Compounds of Tetravalent Molybdenum

AU Sand, J.; Maas, Johanna

CS Chem. Lab. Akad. Wiss., Munchen

SO Berichte der Deutschen Chemischen Gesellschaft (1908), 40, 4504-13

CODEN: BDCGAS; ISSN: 0365-9496

DT Journal

LA Unavailable

AB When a solution of molybdic acid in HCNS is electrolytically reduced (Ber., 38, 3384 and 39, 761) very complex salts are formed. If such a solution is treated with ammonia in excess and an ammoniacal solution of ZnCl_2 , a yellow precipitate of a zinc salt is formed, having the formula $[\text{Mo}(\text{SCN})_6(\text{NH}_3)_4]\text{Zn}$. Most of the compounds mentioned below were made directly or indirectly from this substance. When boiled with dilute acetic acid, the filtrate precipitates crystals of the acetate, $\text{Mo}(\text{SCN})_6(\text{NH}_3)_3(\text{H}_2\text{O})\text{H}_2\text{CH}_3\text{CO}_2\text{H}$. This salt is not easily soluble in acetic acid and is insoluble in benzene and ether. It is soluble in alcohol and in water, in the latter case with an acid reaction. The mother substance of this series can be prepared directly from ammonium molybdate and ammonium thiocyanate. These are dissolved separately in H_2O , treated with fuming HCl and reduced electrolytically. (Raw material.) By washing with ice-cold water, Cl is removed. When dried in a vacuum over H_2SO_4 , this compound, unlike the acetate, turns brown and loses weight. On exposure to air, the original color and weight are restored. By treating the raw material mentioned above in a variety of ways, a number of complex compounds were obtained; the discussion of these is given in the original paper. It was proved that the valence of the Mo in the compounds prepared is four; to establish this point, the Zn salt was heated for several hours with concentrate HCl in a stream of CO_2 and, after cooling, was treated with MnSO_4 and dilute H_2SO_4 . The residue from this treatment was then titrated with KMnO_4 and the unknown valence determined in this way.

CC 6 (Inorganic Chemistry)

IT 7439-98-7, Molybdenum

(comps., of tetravalent Mo)

L33 ANSWER 14 OF 14 HCAPLUS COPYRIGHT 2007 ACS on STN

AN 1906:108642 HCAPLUS

DN 0:108642

TI Action of ammonia and potassium hydroxide on solutions of zinc salts

AU Kuriloff, Basil B.

SO Bulletin de l'Academie Imperiale des Sciences de St.-Petersbourg (1901), 1, 95-103

From: J. Chem. Soc., Abstr. 82, II, 139 1902

CODEN: BUACAN
 DT Journal
 LA Unavailable
 AB Also abstracted in Chemical Centr., 1901, ii, 1222. The excess of ammonia or potassium hydroxide which is necessary to dissolve the precipitate formed when the alkali is added to a solution of zinc chloride or sulphate has been determined, and the results show that in both cases the more dilute the solutions of ammonia or potassium hydroxide, the greater must be the actual proportion of alkali to zinc salt. Solutions weaker than decinormal do not appreciably affect the precipitate. The proportion of alkali required is also greater for solutions of the sulphate than for those of the chloride, but the data obtained for these two solutions are otherwise very similar. No quantitative determinations to show the influence of the chlorine or sulphate ions are given, but the solid phase which is formed when potassium hydroxide acts on zinc sulphate contains sulphate, and this group cannot be removed by washing with water.
 CC 6 (Inorganic Chemistry)

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=> D QUE
 L3 1 SEA FILE=REGISTRY ABB=ON 7646-85-7
 L4 1 SEA FILE=REGISTRY ABB=ON 7732-18-5
 L6 51847 SEA FILE=HCAPLUS ABB=ON L3 OR ZNCL2 OR ZINC CHLORIDE
 L7 2720480 SEA FILE=HCAPLUS ABB=ON L4 OR WATER?
 L34 3655 SEA FILE=WPIX ABB=ON L6 AND L7
 L35 10 SEA FILE=WPIX ABB=ON L34 AND DEWATER?
 L36 216 SEA FILE=WPIX ABB=ON L34 AND REMOV?(4A) (WATER? OR H2O)
 L37 49 SEA FILE=WPIX ABB=ON L36 AND DISTIL?
 L38 8 SEA FILE=WPIX ABB=ON L37 AND ?NITRIL?
 L40 9 SEA FILE=WPIX ABB=ON L36 AND ANHYDROUS?(4A) (ZNCL2 OR ZINC CHLORIDE)

L41 26 SEA FILE=WPIX ABB=ON L35 OR L38 OR L40
 L42 10 SEA FILE=WPIX ABB=ON L37 AND ?AZEOTROP?
 L43 33 SEA FILE=WPIX ABB=ON L41 OR L42
 L44 3 SEA FILE=WPIX ABB=ON L43 AND B01D?/IC
 L45 4 SEA FILE=WPIX ABB=ON L43 AND B01D?/IPC
 L48 4 SEA FILE=WPIX ABB=ON (L44 OR L45)

=> D L48 FULL 1-4

L48 ANSWER 1 OF 4 WPIX COPYRIGHT 2007 THE THOMSON CORP on STN
 AN 2004-156510 [15] WPIX
 DNC C2004-062179 [15]
 TI Removal of water from zinc chloride
 , used as starting material for galvanizing metal or Lewis acid-type
 catalyst, involves distillation after adding aprotic polar
 diluent that has higher boiling point than water or forms
 (hetero)azeotrope
 DC E16; E32; J04; M13
 IN BARTSCH M; BAUMANN M; BAUMANN R; HADERLEIN G; JUNGKAMP T; LUYKEN H;
 SCHEIDEL J
 PA (BADI-C) BASF AG
 CYC 103
 PI WO 2004007371 A1 20040122 (200415)* DE 19[0]
 DE 10231296 A1 20040122 (200416) DE
 AU 2003244640 A1 20040202 (200450) EN
 EP 1521722 A1 20050413 (200525) DE
 BR 2003011998 A 20050426 (200530) PT
 KR 2005023408 A 20050309 (200552) KO
 US 20050207968 A1 20050922 (200563) EN
 MX 2004012177 A1 20050301 (200568) ES
 JP 2005538014 W 20051215 (200582) JA 16
 CN 1668533 A 20050914 (200607) ZH
 EP 1521722 B1 20060405 (200624) DE
 TW 2004007185 A 20040516 (200628) ZH
 DE 50302903 G 20060518 (200637) DE
 ES 2261949 T3 20061116 (200677) ES
 IN 2005CN00162 P4 20070330 (200730) EN
 ADT WO 2004007371 A1 WO 2003-EP7149 20030704; DE 10231296 A1 DE 2002-10231296
 20020710; TW 2004007185 A TW 2003-116404 20030617; AU 2003244640 A1 AU
 2003-244640 20030704; BR 2003011998 A BR 2003-11998 20030704; CN 1668533 A
 CN 2003-816294 20030704; DE 50302903 G DE 2003-502903 20030704; EP 1521722
 A1 EP 2003-738116 20030704; EP 1521722 B1 EP 2003-738116 20030704; DE
 50302903 G EP 2003-738116 20030704; ES 2261949 T3 EP 2003-738116 20030704;
 EP 1521722 A1 WO 2003-EP7149 20030704; BR 2003011998 A WO 2003-EP7149
 20030704; US 20050207968 A1 WO 2003-EP7149 20030704; MX 2004012177 A1 WO
 2003-EP7149 20030704; JP 2005538014 W WO 2003-EP7149 20030704; EP 1521722
 B1 WO 2003-EP7149 20030704; DE 50302903 G WO 2003-EP7149 20030704; JP
 2005538014 W JP 2004-520478 20030704; MX 2004012177 A1 MX 2004-12177
 20041206; US 20050207968 A1 US 2004-519513 20041223; KR 2005023408 A KR
 2005-700471 20050110; IN 2005CN00162 P4 WO 2003-EP7149 20030704; IN
 2005CN00162 P4 IN 2005-CN162 20050210
 FDT DE 50302903 G Based on EP 1521722 A; AU 2003244640 A1 Based on
 WO 2004007371 A; EP 1521722 A1 Based on WO 2004007371 A; BR
 2003011998 A Based on WO 2004007371 A; MX 2004012177 A1 Based on WO
 2004007371 A; JP 2005538014 W Based on WO 2004007371 A; EP 1521722
 B1 Based on WO 2004007371 A; DE 50302903 G Based on WO 2004007371
 A; ES 2261949 T3 Based on EP 1521722 A
 PRAI DE 2002-10231296 20020710
 IC ICM B01D003-34; C01G009-04

ICS B01D003-36; C07C253-34

IPCI B01D0003-34 [I,C]; B01D0003-34 [I,C];
B01D0003-36 [I,A]; B01D0003-36 [I,A]; C01G0009-00 [I,C];
C01G0009-00 [I,C]; C01G0009-04 [I,A]; C01G0009-04 [I,A]; C07C0253-00
[I,C]; C07C0253-00 [I,C]; C07C0253-34 [I,A]; C07C0253-34 [I,A]

IPCR B01D0003-34 [I,C]; B01D0003-36 [I,A]; C01G0009-00
[I,C]; C01G0009-04 [I,A]; F26B0005-00 [I,A]; F26B0005-00 [I,C];
F26B0007-00 [I,A]; F26B0007-00 [I,C]

AB WO 2004007371 A1 UPAB: 20060203

NOVELTY - Removal of water from mixture (I) containing zinc chloride (ZnCl₂) comprises (1) treating (I) with aprotic, polar diluent (II) that (a) forms no azeotrope with water, boils above the boiling point (b.pt.) of water at the distillation pressure and is liquid at this b.pt. of water or (b) forms a (hetero) azeotrope (III) with water at the distillation pressure and temperature; and (2) distillation to separate water or (III), giving an anhydrous mixture containing ZnCl₂ and (II).

USE - Anhydrous zinc chloride (ZnCl₂) and anhydrous mixtures of a liquid diluent and ZnCl₂ are valuable starting materials for galvanizing metals or catalysts or catalyst constituents with Lewis acid properties, e.g. in hydrocyanation of 3-pentenitrile in the presence of nickel(0) phosphite catalyst.

ADVANTAGE - Recovery of zinc chloride (ZnCl₂) usually gives mixtures containing water. Existing methods of recovering anhydrous ZnCl₂ from aqueous solutions are uneconomical, give an impure product or are either ineffective or hazardous. The present process is technically simple and economical. It is suitable for discontinuous or continuous operation.

TECH INORGANIC CHEMISTRY - Preferred Mixture: Mixture (I) containing water and zinc chloride (ZnCl₂) has pH less than 7 and down to 0 and is treated with an acid, preferably hydrochloric acid (HCl).

ORGANIC CHEMISTRY - Preferred Diluents: Diluent (II) is an organic diluent, preferably containing nitrile group(s), especially an aliphatic, saturated or olefinically unsaturated nitrile. The diluent forming a (hetero)azeotrope with water preferably is an aliphatic, olefinically unsaturated nitrile selected from 2- and 3- and 4-cis- and -trans-pentenitriles, 4-pentenitrile, E- and Z-2-methyl-2-butenitriles, 2-methyl-3-butenitrile and mixtures of these.

ABEX EXAMPLE - The process was carried out continuously in a vacuum distillation column (inside diameter 50 mm, height 130 cm) with metal fabric packing. This had a thin film evaporator as reboiler, top condenser operating at 30degreesC and phase separation vessel cooled to 0degreesC in the recycling system. 240 g/hour solution of 30 wt.% zinc chloride in trans-3-pentenitrile (IIA), with a water content of 0.4 wt.%, were introduced above the packing. Distillation at 100 kPa (absolute) and 344 K gave a 2-phase mixture in the condenser. The upper phase, essentially (IIA), was recycled to the top of the column. The lower phase, essentially water, was pumped away continuously. A homogeneous solution of ZnCl₂ in (IIA) was separated at 348 K from the reboiler. This had a water content of 76 ppm weight after distillation for 17 hours and 50 ppm weight after 41 hours.

FS CPI

MC CPI: E11-Q01; E35-C; J04-E04; M13-A02

L48 ANSWER 2 OF 4 WPIX COPYRIGHT 2007

THE THOMSON CORP on STN

AN 2002-731291 [79] WPIX

DNC C2002-207116 [79]

TI Chromium-based catalyst composition for polymerizing conjugated dienes, comprises combination of or reaction product of ingredients comprising chromium-containing compound, organomagnesium compound and silyl phosphonate

DC A12; E11; E12

IN LUO S

PA (BRID-C) BRIDGESTONE CORP

CYC 21

PI US 20020115558 A1 20020822 (200279)* EN 10[0]

US 6465585 B2 20021015 (200279) EN

WO 2002066525 A1 20020829 (200279) EN

ADT US 20020115558 A1 US 2001-788795 20010219; WO 2002066525 A1 WO 2002-US4789 20020218

PRAI US 2001-788795 20010219

IPCR B01J0023-16 [N,C]; B01J0023-26 [N,A]; B01J0031-02 [I,A]; B01J0031-02 [I,C]; B01J0031-12 [I,A]; B01J0031-12 [I,C]; C08F0136-00 [I,C]; C08F0136-06 [I,A]; C08F0004-00 [I,C]; C08F0004-69 [I,A]

AB US 20020115558 A1 UPAB: 20060120

NOVELTY - A chromium-based catalyst composition comprises a mixture or a reaction product of chromium-containing compound, an organomagnesium compound and a silyl phosphonate.

DETAILED DESCRIPTION - An INDEPENDENT CLAIM is included for formation of conjugated diene polymers which involves polymerizing conjugated diene monomers in presence of the chromium-based catalyst composition.

USE - For polymerizing conjugated dienes such as 1,3-butadiene into polymers such as syndiotactic 1,2-polybutadiene. The syndiotactic 1,2-polybutadiene is used for manufacturing films and packaging materials and in many molding applications.

ADVANTAGE - The chromium-based catalyst composition does not contain carbon disulfide, therefore the toxicity, objectionable smell, dangers and expense associated with the use of carbon disulfide are eliminated. The chromium-containing compounds are stable, inexpensive and readily available. The chromium-based catalyst composition has high catalytic activity in a wide variety of solvents including the environmentally-preferred non-halogenated solvents such as aliphatic and cycloaliphatic hydrocarbons.

The syndiotactic 1,2-polybutadiene produced using the catalyst composition has higher melting temperature and higher syndiotacticity than those produced conventionally. The green strength of elastomers, particularly in tires can be improved by incorporating the syndiotactic 1,2-polybutadiene polymers. The incorporation of syndiotactic 1,2-polybutadiene into rubber compositions that are used in the supporting carcass of tires prevents or minimizes the distortion of tires during building and curing procedures. The incorporation of the syndiotactic polymers into tire tread compositions reduces the heat build-up and improves the wear and tear resistance of the tire treads. Polymers having desirable properties are obtained by using narrower range of catalyst compositions and catalyst ingredients ratios.

TECH ORGANIC CHEMISTRY - Preferred Compounds: The organomagnesium compound is of formula (I) or (II).

MgR₁₂ (I)R₂MgX (II)

R₁ = mono-valent organic group that is attached to magnesium atom via carbon atom, preferably alkyl, (un)substituted cycloalkyl, alkenyl, (un)substituted cycloalkenyl, (un)substituted aryl, aralkyl, alkaryl, allyl or alkynyl;

R2 = R1; and

X = hydrogen, halogen, carboxylate, alkoxide, or aryloxide.

The organomagnesium compound is preferably hydrocarbylmagnesium hydride, hydrocarbylmagnesium halide, hydrocarbylmagnesium carboxylate, hydrocarbylmagnesium alkoxide or hydrocarbylmagnesium aryloxide. The conjugated diene monomers are 1,3-butadiene.

Preferred Silyl Phosphonate: The silyl phosphonate is an acyclic silyl phosphonate of formula (III) or cyclic silyl phosphonate of formula (IV). R3, R4 = hydrogen or R1; and

R5 = bond between silicon atoms or divalent organic group, preferably (un)substituted alkylene, (un)substituted cycloalkylene, (un)substituted alkenylene, (un)substituted cycloalkenylene, or (un)substituted arylene.

The chromium-containing compound is selected from chromium organophosphate, chromium organophosphonate, chromium organophosphinate, chromium alkoxide or aryloxide, or organochromium compound.

Preferred Composition: The molar ratio of the organomagnesium compound to chromium-containing compound is 1:1-50:1, preferably 2:1-30:1, and the molar ratio of the silyl phosphonate to chromium-containing compound is 0.5:1-50:1, preferably 1:1-25:1. During the formation of conjugated diene polymers, the catalyst composition is present in an amount of 0.01-2 mmol/100 g of monomer.

Preferred Process: The chromium-based catalyst composition is formed by combining chromium-containing compound, organomagnesium compound and silyl phosphonate, in order.

INORGANIC CHEMISTRY - Preferred Compounds: The chromium-containing compound is selected from a chromium carboxylate, chromium halide, chromium pseudo-halide or chromium oxyhalide.

ABEX SPECIFIC COMPOUNDS - The organomagnesium compound is specifically claimed as dimethylmagnesium, diethylmagnesium, di-n-propylmagnesium, diisopropylmagnesium, di-n-butylmagnesium, di-sec-butylmagnesium, diisobutylmagnesium, di-t-butylmagnesium, di-n-hexylmagnesium, di-n-octylmagnesium, diphenylmagnesium, di-p-tolylmagnesium, or dibenzylmagnesium. - The acyclic silyl phosphonate is specifically claimed as bis(triethylsilyl) phosphonate, bis(trimethylsilyl) phosphonate, bis(tri-n-propylsilyl)phosphonate, bis(triisopropylsilyl)phosphonate, bis(tri-n-butylsilyl)phosphonate, bis(tricyclohexylsilyl)phosphonate, bis(triphenylsilyl)phosphonate, bis(tris(2-ethylhexyl)silyl)phosphonate, or bis(tris(2,4,6-trimethylphenyl)silyl)phosphonate. - The cyclic silyl phosphonate is selected from 10 specific compounds such as 2-oxo-(2H)-4,5-disila-1,3,2-dioxaphosphorane, and 2-oxo-(2H)-4,5-disila-4,4,5,5-tetramethyl-1,3,2-dioxaphosphorane. - The chromium-containing compound is specifically claimed as chromium carbamate, chromium dithiocarbamate, or chromium xanthate.

EXAMPLE - Anhydrous phosphorus acid (in grams) (22.1), hexaethyldisiloxane (99.5), anhydrous zinc chloride (1.33) and toluene (230 ml) were mixed. The reaction mixture was heated to reflux for 29 hours with continuous removal of water via the Dean-Stark trap. The toluene solvent and the unreacted hexaethyldisiloxane were removed by distillation at atmospheric pressure. The remaining crude product was distilled under vacuum and bis(triethylsilyl) phosphonate was produced as a colorless liquid. The yield of the product was 81 %. An oven-dried glass bottle was purged thoroughly with nitrogen gas and was charged with a blend (236) of 1,3-butadiene/hexanes containing 21.2 weight % of 1,3-butadiene. To the glass bottle, dibutylmagnesium (0.90 mmol), chromium (III) 2-ethylhexanoate (0.15 mmol), obtained bis(trimethylsilyl)phosphonate (0.6 mmol) were added. The bottle was heated in a water bath maintained at 50 degrees C for 15 hours. The polymerization was terminated by addition of isopropanol (10 ml) containing 2,6-di-tert-butyl-4-methylphenol (1). The polymerized mixture was coagulated with isopropanol

(3 liters). The resulting syndiotactic 1,2-polybutadiene was isolated by filtration and was dried to a constant weight under vacuum at 60 degrees C. The polymer had a melting temperature of 150 degrees C as measured by differential scanning calorimetry. The 1H and 13C nuclear magnetic resonance (NMR) analysis indicated a 1,2-linkage content of 78 % and a syndiotacticity of 79 %.

FS CPI

MC CPI: A02-A06D; A04-B01A; E05-B01; E05-E01; E05-E02B; E05-G; E05-L03A; E31-C; E35-P

L48 ANSWER 3 OF 4 WPIX COPYRIGHT 2007 THE THOMSON CORP on STN

AN 2000-303179 [26] WPIX

DNC C2000-091888 [26]

TI Method of dewatering difficult sludges comprises a three-compartment treatment with an inorganic coagulant, a microparticle and finally with a flocculant

DC A14; A97; D15

IN SHAH J; SOMMESE A G; SOMMESE G

PA (NALC-C) NALCO CHEM CO

CYC 85

PI WO 2000017112 A1 20000330 (200026) * EN 17[0]

AU 9948457 A 20000410 (200035) EN

US 6083404 A 20000704 (200036) EN

NO 2001001367 A 20010509 (200134) NO

EP 1115663 A1 20010718 (200142) EN

JP 2002526253 W 20020820 (200258) JA 16

TW 509663 A 20021111 (200353) ZH

NZ 510317 A 20031219 (200404) EN

DE 69933300 E 20061102 (200675) DE

DE 69933300 T2 20070104 (200705) DE

ES 2273503 T3 20070501 (200731) ES

ADT WO 2000017112 A1 WO 1999-US14750 19990629; US 6083404 A US 1998-156820 19980918; AU 9948457 A AU 1999-48457 19990629; DE 69933300 E DE 1999-633300 19990629; DE 69933300 T2 DE 1999-633300 19990629; EP 1115663 A1 EP 1999-932065 19990629; DE 69933300 E EP 1999-932065 19990629; DE 69933300 T2 EP 1999-932065 19990629; NZ 510317 A NZ 1999-510317 19990629; NO 2001001367 A WO 1999-US14750 19990629; EP 1115663 A1 WO 1999-US14750 19990629; JP 2002526253 W WO 1999-US14750 19990629; NZ 510317 A WO 1999-US14750 19990629; DE 69933300 E WO 1999-US14750 19990629; DE 69933300 T2 WO 1999-US14750 19990629; TW 509663 A TW 1999-113120 19990731; JP 2002526253 W JP 2000-574029 19990629; NO 2001001367 A NO 2001-1367 20010316; ES 2273503 T3 EP 1999-932065 19990629

FDT DE 69933300 E Based on EP 1115663 A; DE 69933300 T2 Based on EP 1115663 A; AU 9948457 A Based on WO 2000017112 A; EP 1115663 A1 Based on WO 2000017112 A; JP 2002526253 W Based on WO 2000017112 A; NZ 510317 A Based on WO 2000017112 A; DE 69933300 E Based on WO 2000017112 A; DE 69933300 T2 Based on WO 2000017112 A; ES 2273503 T3 Based on EP 1115663 A

PRAI US 1998-156820 19980918

IC ICM C02F001-52; C02F011-14

IPCI C02F0001-52 [I,A]; C02F0001-52 [I,A]; C02F0001-52 [I,C]; C02F0001-52 [I,C]; C02F0001-54 [I,A]; C02F0001-54 [I,A]; C02F0001-54 [I,C]; C02F0001-56 [I,A]; C02F0001-56 [I,A]; C02F0011-02 [I,A]; C02F0011-02 [I,A]; C02F0011-02 [I,C]; C02F0011-14 [I,A]; C02F0011-14 [I,A]; C02F0011-14 [I,C]

IPCR B01D0021-01 [I,A]; B01D0021-01 [I,C]; C02F0011-14 [I,A]; C02F0011-14 [I,C]

AB WO 2000017112 A1 UPAB: 20060116

NOVELTY - The addition of microparticle component in a three-compartment treatment for dewatering of difficult sludges increases the

amount of free drainage. The method incorporates a three-component treatment and comprises dosing the sludge initially with an inorganic coagulant (A), then dosing the sludge inorganic mixture with a microparticle (B) and final dosing with a high molecular weight flocculant.

USE - To dewater difficult sludges, especially sludges derived from an autoheated thermophilic aerobic digestion system.

ADVANTAGE - The method has ability to produce strong dewaterable floc in difficult sludge that can readily release water and can withstand the shear of mechanical dewatering. It also has the ability to cost effectively dewater difficult sludges by producing strong floc formation at dosages lower than those often required in prior art.

TECH POLYMERS - Preferred Microparticle: (B) consists of an organic polymer with a molecular weight of less than one million amu and a colloidal inorganic material(c).

Preferred Flocculant: The flocculant has a molecular weight of greater than one million amu and is anionic, cationic and non-charged. The anionic flocculant consists of polyacrylates, poly(meth)acrylates, acrylamide/sodium acrylate copolymers, acrylamide/acrylamido-propylsulfonic acids copolymers and terpolymers of acrylamide/acrylamido-propylsulfonic acid/sodium acrylate. The cationic flocculant consists of poly(dimethylaminoethylmethacrylate methyl chloride salt) (DMAEM.MCQ), poly(dimethylaminoethylacrylate methyl chloride salt) (DMAEA.MCQ), acrylamide/DMAEA.MCQ copolymers, acrylamide/diallyldimethylammonium chloride(DADMAC) copolymers, acrylamide/DADMAC/DMAEA.MCQ terpolymers, AcAm/dimethylaminoethylacrylate benzyl chloride salt(DMAEA.BCQ)/DMAEA.MCQ terpolymers, copolymers of vinylamine/vinylformamide. The non-charged flocculant consists of polyacrylamides, polyvinyl pyrrolidone, polyvinylforamides and polyacrylic acid.

INORGANIC CHEMISTRY - Preferred Coagulant: (A) consists of FeCl₃, FeSO₄, AlCl₃, Alum, Al₂Cl_x(H₂O)_y, ZnCl₂, ZnCl₄ and polyaluminum chloride.

Preferred Colloidal Material: (c) consists of colloidal silica, colloidal zinc, colloidal aluminum, colloidal borosilicate, betonites, hectorites, smectites, colloidal aluminas and colloidal zincs.

ENVIRONMENT - Preferred Sludge: The sludge is derived from a autothermal thermophilic aerobic digestion system.

ABEX EXAMPLE - To an autothermal thermophilic aerobic digestion (ATAD) sludge was added 5000 ppm of alum. This sludge was then treated with Nalco 7194 plus (RTM: Cationic Charged Flocculant) and Nalco 7139 plus (RTM: Cationic Charged flocculant) at various dosages. The free drainage (ml H₂O/10 sec) for Nalco 7194 plus at 350, 450 550(ppm) dosage was 22, 32 and 28, while the free drainage for Nalco 7139 plus was 15, 20 and 25 respectively. The control sludge was treated with Nalco 8677 plus (RTM; Organic Microparticle) at different dosages levels, keeping flocculant constant. The free drainage was then found to be 58, 60, 66 for Nalco 7194 plus at 450(ppm) and 64, 68 and 72 for Nalco 7139 plus at 450(ppm) then microparticle dosage was 150, 250 and 350 ppm respectively. Thus almost a four fold increase in drainage was observed when the microparticle was used.

FS CPI

MC CPI: A12-M01; A12-M02; A12-W11E; D04-A01B

L48 ANSWER 4 OF 4 WPIX COPYRIGHT 2007

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AN 1999-540689 [45] WPIX

DNC C1999-157934 [45]

DNN N1999-400749 [45]

TI Ion conductive matrixes for forming membranes, composite electrode, electrochemical cell, fuel cell and water electrolizer

DC A32; A85; E16; E36; E37; J03; L03; P56; X16
 IN DUVDEVANI T; MELMAN A; PELED E
 PA (UYRA-N) UNIV RAMOT APPLIED RES & IND DEV LTD; (UYTE-N) UNIV TEL AVIV
 FUTURE TECHNOLOGY DEV LP

CYC 82

PI WO 9944245 A1 19990902 (199945)* EN 35[2]

AU 9926369 A 19990915 (200004) EN

EP 1066656 A1 20010110 (200103) EN

IL 123419 A 20001206 (200103) EN

IL 126830 A 20010520 (200153) EN

KR 2001034536 A 20010425 (200164) KO

JP 2002505506 W 20020219 (200216) JA 41

US 6811911 B1 20041102 (200472) EN

KR 573655 B1 20060426 (200724) KO

ADT WO 9944245 A1 WO 1999-IL109 19990222; IL 123419 A IL 1998-123419 19980224;
 IL 126830 A IL 1998-126830 19981030; AU 9926369 A AU 1999-26369 19990222;
 EP 1066656 A1 EP 1999-906424 19990222; EP 1066656 A1 WO 1999-IL109
 19990222; JP 2002505506 W WO 1999-IL109 19990222; US 6811911 B1 WO
 1999-IL109 19990222; JP 2002505506 W JP 2000-533910 19990222; KR
 2001034536 A KR 2000-709294 20000823; US 6811911 B1 US 2000-622676
 20001018; KR 573655 B1 WO 1999-IL109 19990222; KR 573655 B1 KR 2000-709294
 20000823

FDT AU 9926369 A Based on WO 9944245 A; EP 1066656 A1 Based on
 WO 9944245 A; JP 2002505506 W Based on WO 9944245 A; US
 6811911 B1 Based on WO 9944245 A; KR 573655 B1 Previous
 Publ KR 2001034536 A; KR 573655 B1 Based on WO 9944245 A

PRAI IL 1998-126830 19981030

IL 1998-123419 19980224

IC ICM H01M004-58; H01M008-02; H01M008-10

ICS A61K009-14; B23P019-00; C08J005-20; C25B011-04; C25B013-00;

H01B001-06; H01M004-32; H01M004-42; H01M004-50; H01M004-86; H01M006-00

ICA H01M006-18

IPCI H01M0004-58 [I,A]; H01M0004-58 [I,C]

IPCR B01D0071-00 [I,C]; B01D0071-02 [I,A]; B23P0019-00

[I,A]; B23P0019-00 [I,C]; C08J [I,S]; C08J0005-20 [I,A]; C08J0005-20

[I,C]; C08J0005-22 [I,A]; C25B0011-00 [I,C]; C25B0011-04 [I,A];

C25B0013-00 [I,A]; C25B0013-00 [I,C]; C25B0013-04 [I,A]; H01B0001-06

[I,A]; H01B0001-06 [I,C]; H01M [I,S]; H01M0012-00 [I,C]; H01M0012-06

[I,A]; H01M0002-16 [I,A]; H01M0002-16 [I,C]; H01M0004-04 [I,A];

H01M0004-04 [I,C]; H01M0004-32 [I,A]; H01M0004-32 [I,C]; H01M0004-42

[I,A]; H01M0004-42 [I,C]; H01M0004-50 [I,A]; H01M0004-50 [I,C];

H01M0004-58 [I,A]; H01M0004-58 [I,C]; H01M0004-86 [I,A]; H01M0004-86

[I,C]; H01M0006-00 [I,A]; H01M0006-00 [I,C]; H01M0006-04 [I,A];

H01M0006-04 [I,C]; H01M0006-06 [I,A]; H01M0006-18 [I,A]; H01M0006-18

[I,C]; H01M0008-02 [I,A]; H01M0008-02 [I,C]

AB WO 1999044245 A1 UPAB: 20050705

NOVELTY - The ion conductive matrix comprises 5 - 60 volume percent
 (volume%) of inorganic powder in form of sub-micron particles having good
 aqueous electrolyte absorption capacity, 5 - 50 volume% of polymeric binder
 compatible with an aqueous electrolyte, and 10 - 90 volume% of an aqueous
 electrolyte.

DETAILED DESCRIPTION - INDEPENDENT CLAIMS are also included for:

(i) Method for casting membrane which comprises preparing mixture
 comprising inorganic powder, polymeric binder, at least one high boiling
 point solvent with boiling point above 100 degreesC and at least one low
 boiling point solvent in which the polymeric binder is soluble or forms a
 gel at casting temperature. Film is casted out of mixture and low boiling
 point solvent is evaporated from mixture to form solid film. Solid film is
 washed to replace high boiling point solvent with aqueous electrolyte
 solution. Alternatively, mixture is heated to its softening temperature

and film is formed by hot extrusion of softened mixture. The high boiling point solvent used in the mixture has boiling point above 90 degreesC. Film is cooled to obtain solid film, and washed to replace solvent with aqueous electrolyte solution.

(ii) Method for casting composite electrode comprising steps involved in casting membrane. Alternatively, preparing composite electrode by extrusion which comprises steps involved in preparing membrane by extrusion.

USE - For forming membranes, composite electrode, electrochemical cell, fuel cell and water electrolyzer.

ADVANTAGE - Novel, low cost and highly conductive ion conducting matrix, membranes and electrodes are provided. The ion conducting membranes have good porosity and mechanical properties. Internal lubricants with low solubility in water is used to achieve solubility factor not higher than 14 (cal/cc)^{1/2}, thereby preventing the migration of internal lubricants out of ion conductive membranes when they come in contact with water at washing phase or acid loading phase.

TECH INORGANIC CHEMISTRY - Preferred Matrix: The ion conducting matrix is a proton conducting matrix and comprises desirably 5 - 50 % of inorganic powder such as silicon dioxide (SiO₂), zirconium oxide (ZrO₂), boron trioxide (B₂O₃), titanium oxide (TiO₂), aluminum oxide (Al₂O₃), and/or optional hydroxides or oxy-hydroxides of Ti, Al, B or Zr with a surface area of at least 10 m²/g. The matrix optionally comprises 0.1 - 25 % of nonvolatile liquid lubricant which is compatible with all the components in matrix.

Preferred Electrolyte: The aqueous electrolyte consists of aqueous soluble salt and/or base which is used in aqueous solution having molar concentration of 0.1 - 10 M, preferably 1 - 5 M.

Alkali metal salts, alkali earth metal salts, R₄NX, where

R = organic radical;

X = anion derived from an inorganic acid.

Ammonium chloride (NH₄Cl) and/or zinc chloride (

ZnCl₂) is used as the aqueous soluble salt.

R₄NOH, where

R = hydrogen or an organic radical, alkali and/or alkali earth base compound is used as the aqueous soluble bases.

Preferred Membrane: The membrane comprises ion conducting matrix having electronically nonconductive inorganic material with particle size less than 150 nm. The membrane comprises pores with size less than 50 nm. The inorganic powder of matrix is treated with acid or base prior to preparation of membrane. The membrane further comprises electronic nonconductive reinforcing element.

Preferred Electrode: The composite electrode comprises 10 - 70 vol.% of the matrix and remaining electrode material.

Preferred Electrochemical Cell: The electrochemical cell comprises membrane or at least one electrode having electrode material of carbon and/or graphite, metal oxides such as RuO₂, WO_x or MnO₂. Cadmium, zinc, and/or aluminum or its alloys is used as anode active material. Manganese oxide (MnO₂), silver oxide or nickel oxy hydroxide (NiOOH) is used as cathode active material. Zn or Al anode and oxygen or air electrode which consists of double layer film with hydrophobic air side and hydrophilic ionic membrane side is used. The air electrode catalyst is compatible with aqueous solutions of ionic conductive membrane such as oxides of platinum, palladium, gold, silver, copper, manganese, tungsten and/or metal-porphyrin complexes of their salts. The electrochemical cell is single structure unit manufactured by hot pressing the electrodes on both sides of the membrane.

ORGANIC CHEMISTRY - Preferred Lubricant: Diesters of aliphatic or aromatic dibasic acids, esters of phosphoric acids, hydrocarbons or synthetic

hydrocarbons, silicone oils and/or fluorocarbons is used as the lubricant. Preferred Acid: The proton conducting matrix comprises 10 - 90 vol.% of an acid such as $\text{CF}_3(\text{CF}_2)_n\text{SO}_3\text{H}$, $\text{HO}_3\text{S}(\text{CF}_2)_n\text{SO}_3\text{H}$, where $n = 0 - 9$, especially 0 - 4.

sulfuric acid, hydrochloric acid, hydrobromic acid, phosphoric acid and/or nitric acid. The acid is used in an aqueous solution having a molar concentration of 10 - 99 %, preferably 25 - 99 %.

Preferred Solvent: The high boiling point solvent used for casting or preparing membrane or composite electrode is water soluble solvent. Propylene carbonate, ethylene carbonate, dimethyl phthalate, diethyl phthalate, and/or dibutyl phthalate is used as high boiling point solvent for casting or preparing membrane. Tetrahydrofuran, dimethylether (DME), cyclopentanone, acetone, N-methyl pyrrolidone, dimethylacetamide, methylethylketone, and/or dimethyl-formamide is used as the low boiling point solvent for casting or preparing the membrane. Propylene carbonate, diethyl carbonate, dimethyl carbonate, butyrolactone, methyl isoamyl ketone, cyclohexanone, dialkyl phthalate, and/or glycerol triacetate is used as solvent for casting or preparing composite electrode.

POLYMERS - Preferred Binder: Polyvinylidene fluoride, polyvinylidene fluoridehexafluoropropylene, poly(tetrafluoroethylene), poly(methylmethacrylate), polysulfone amide, poly(acrylamide), polyvinyl chloride, poly(acrylonitrile), and/or polyvinyl fluoride is used as the polymeric binder.

ABEX EXAMPLE - The membrane film was manufactured by mixing 0.17 g (24.5 vol.%) of powdered Kynar PVDF 2801-00, 99.8 % (0.5 vol.%) of silicon (IV) oxide with 0.147 g and particle size of 400 m2/g, 20 ml of cyclopentanone and 0.48 ml of propylene carbonate (PC) to obtain viscous mixture. The viscous mixture was poured onto Teflon plate and dried at room temperature for 24 hours to obtain transparent film. The film was washed with double distilled water to remove PC and was found to have porosity of 75 vol.%. The ion conductive membrane was found to have very good mechanical properties.

FS CPI; GMPI; EPI

MC CPI: A09-A03; A12-E06B; A12-E09; A12-E14; E10-A09B8; E10-A22G; E31-A02; E31-B03C; E31-B03D; E31-D01; E31-F05; E31-H05; E31-K05A; E31-P03; E32-A04; E33; E34; E35; J03-A; L03-A02; L03-E01B9; L03-E04B
EPI: X16-A; X16-A02; X16-E01C; X16-E01C1; X16-E05; X16-E06; X16-E09

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